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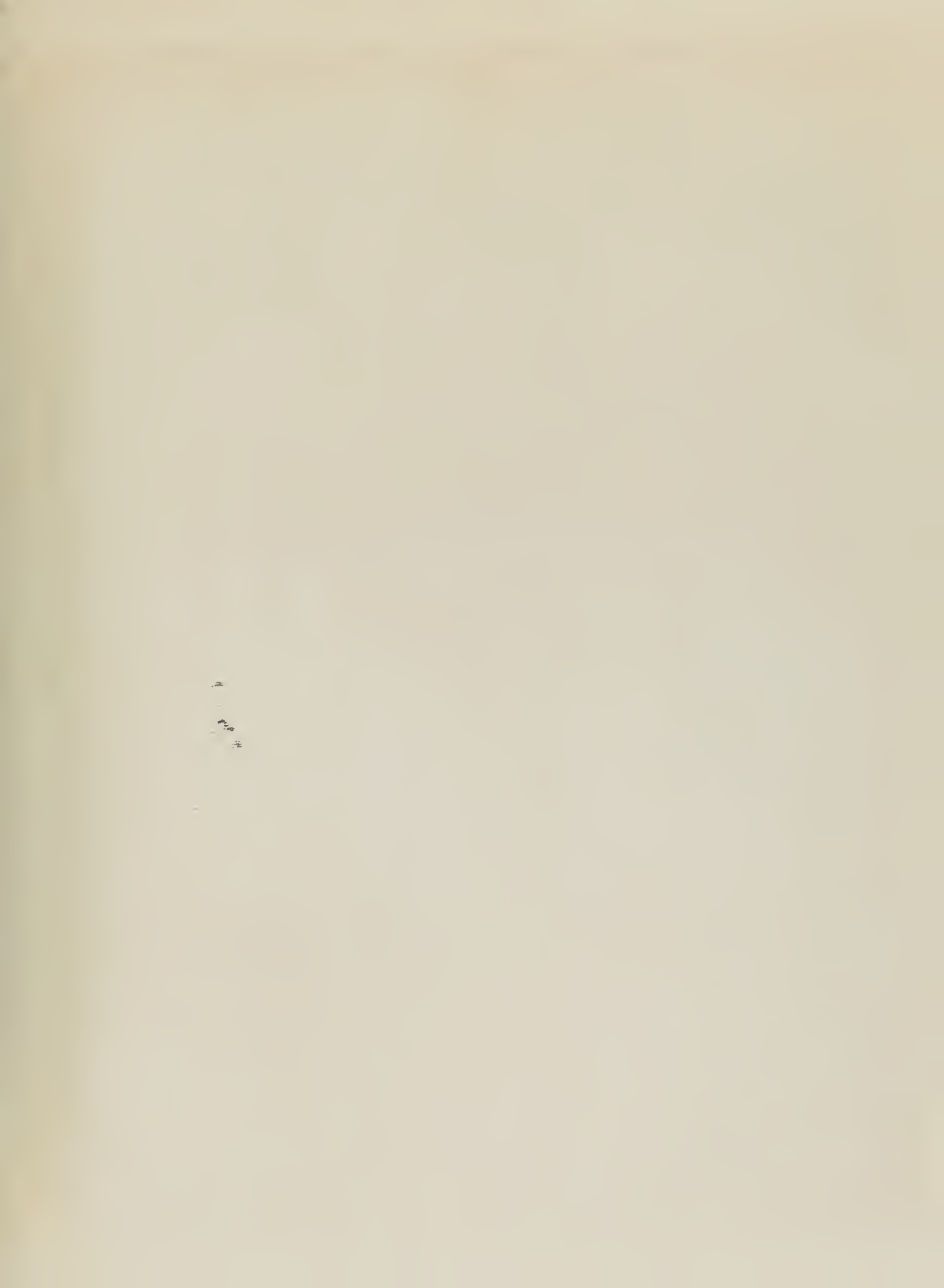
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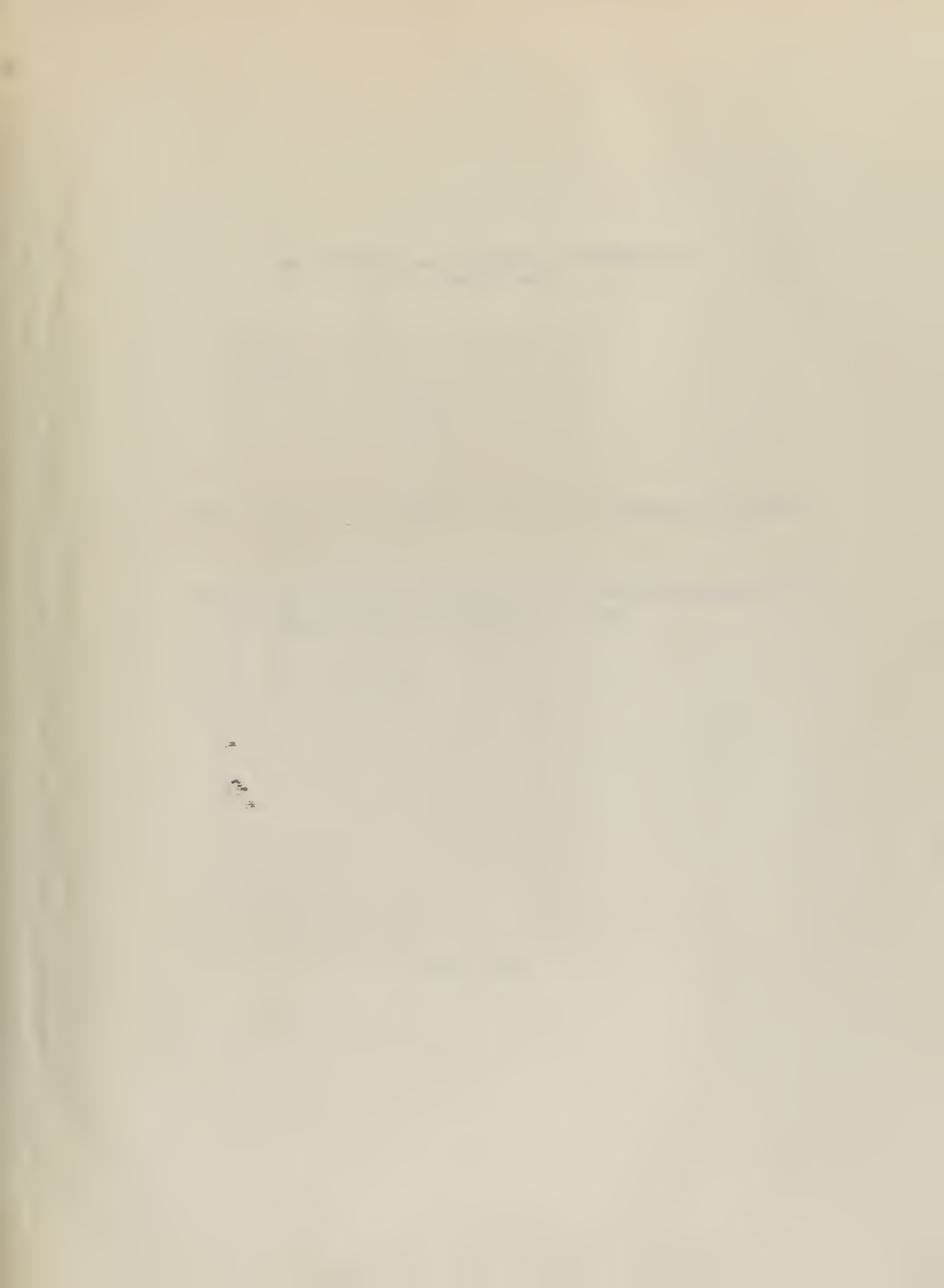
THE USE OF X-RAY PATTERNS
IN DETERMINING SOIL CONSTITUENTS

R. E. Moffat





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THE USE OF X-RAY PATTERNS IN DETERMINING
SOIL CONSTITUENTS

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THESIS

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DETERMINING SOIL CONSTITUENTS

R. E. Moffat

Submitted to the Faculty of Rensselaer Polytechnic Institute

in

Partial Fulfillment of the requirements for the Degree of
Master of Civil Engineering

by

R.E. Moffat

"

May 1954

Thesis

M 66

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To Doctor Arthur A. Burr, of the Department of Metallurgy, the author wishes to express his appreciation for the generosity with which he made available the x-ray and photographic apparatus and facilities.

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the author wishes to express his deep appreciation for his invaluable assistance and advice throughout the work and especially for his active work in making the photographic film records of x-ray diffraction patterns that form a part of the data for this thesis.

SECTION I

INTRODUCTION

OBJECTIVE: This thesis was entered into in order to evaluate a quick and, it is hoped, accurate means for the identification of clay minerals by x-ray diffraction. This evaluation is accomplished by the use of standard samples.

In the literature regarding the field of soil mechanics, x-ray diffraction analysis is often mentioned as a means of identifying soil constituents, especially among the clay minerals. This is possible because clay minerals have been found to have an entirely crystalline structure (7). Therefore, by bombarding a clay mineral with x-rays we may record a diffraction pattern. This pattern is unique for each clay mineral and allows us to "fingerprint" the mineral for identification purposes.

Previous investigations at R.P.I. (2,3) have been concerned with the technique of preparing the specimens x-ray analysis. All used a film strip to record the diffraction pattern. It is felt that this method is rather slow since the average exposure takes three hours, the developing takes another hour and then the film must be dried before evaluation of results can be started.

The installation of a Norelco high-angle goniometer and x-ray spectrometer in the Metallurgy Department of R.P.I. suggested a more rapid means of obtaining the required x-ray diffraction patterns. This machine only requires eighty minutes for an exposure and at the end of that time furnishes a recorded pattern that is ready to work on without any developing or drying time being needed. It is considered that this

method produces a record that can be more easily and accurately evaluated than the film strips. It is estimated that this machine saves about one days time for each specimen tested. The time saving alone would be a strong recommendation for the use of this machine in commercial evaluation work even without the increased accuracy. Both advantages together seem to make the machine an extremely desirable tool.

Since the amount and nature of clay minerals in any soil is a major contributing factor to the physical properties of the soil mass, a quick means of qualitatively determining the identity of the clay fraction is greatly to be desired. The quantitative determination, by weight, of the amount of clay fraction in a given soil mass and its grain-size distribution can be readily attained by hydrometer and sieve analyses.

It was felt that, if the x-ray spectrometer is as good in the final evaluation as it appears to be at first glance, it will furnish a qualitative analytical tool that is just as easy to use as the quantitative methods mentioned above.

SECTION II

APPARATUS

The following apparatus was used in connection with the work in this thesis:

IONIC SATURATION EQUIPMENT

A DUMORE high speed mechanical stirring apparatus was used instead of hand stirring, so that maximum dispersion and ionic saturation of the sample in suspension could be obtained in a minium of time. (See Fig. 1)

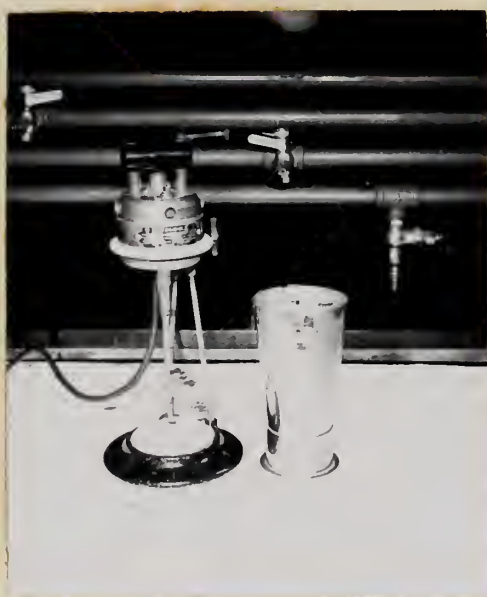


Figure 1
MECHANICAL STIRRING APPARATUS



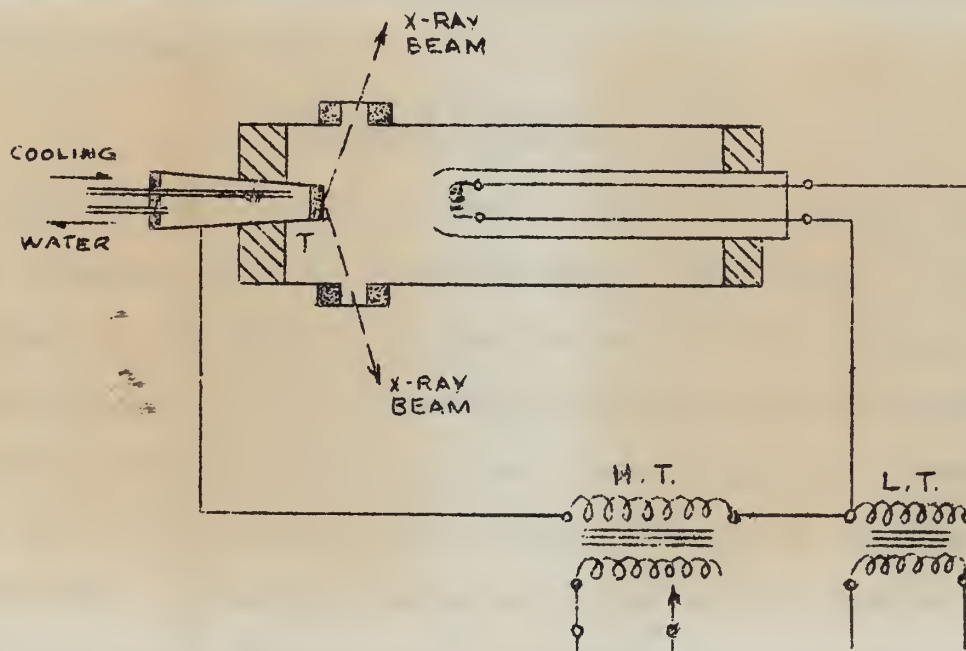
Figure 2
INTERNATIONAL CENTRIFUGE

An International, size 1, type C centrifuge was used to precipitate the clay particles after complete dispersion and ionic saturation had taken place. The only other method would require settlement for many days at normal gravity. The centrifuge produces close to 1000 gravities

and the entire settling process only takes about ten minutes. It was also used in other parts of the work where rapid precipitation was required. (See Fig. 2)

X-RAY TUBE (2)

A Phillips Standard, line-focus, four port x-ray tube with a chromium target was used as a source of x-rays. It consists of an evacuated tube containing two electrodes. These are the cathode which acts as a source of electrons and the anode or target. A high potential difference is applied across the electrodes by means of an external power source.



X-RAY TUBE SCHEMATIC

Figure 3

Under the influence of the electrostatic field thus generated, electrons leave the cathode and strike the anode with high velocity. The character of the x-rays so produced depends in part on the material of

which the target is made. Only a small part of the energy of the electrons is converted into x-rays upon striking the target. The major portion is converted into heat, which is commonly dissipated by means of a water cooling system. A simplified sketch of a typical tube is shown in Figure 3.

FILTERS

No filter was used on the x-ray tube. With a copper target tube it is customary to use a nickel filter to screen out undesirable radiation but with the chromium target used in this work it was not considered necessary to use a filter.

EQUIPMENT PROTECTION

When a water cooled x-ray tube is used, it is absolutely essential that a constant supply of circulating water be maintained. If this is not done, the tube will quickly burn out and since the replacement cost of a single tube is in the neighborhood of 600 dollars the equipment protection question is very important. To help prevent an accidental burn out, a mercury switch device has been installed in the power feed to the tube. The switch is maintained in the on position by the flow of cooling water. Nevertheless it is important to check the flow of cooling water visually each time the tube is used, just in case the switch should be faulty. The cooling system pressure should be sufficient to prevent small fluctuations of the building water supply interrupting the radiation output.

PERSONNEL PROTECTION

Portable lead shields should always be carefully placed to protect operating personnel and others nearby from the harmful effects of scatter radiation.

THE GONIOMETER

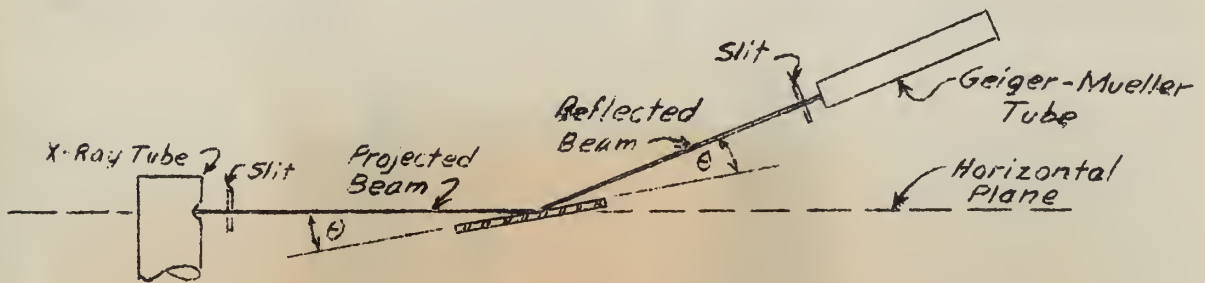
The Norelco high angle goniometer was used to expose the specimens of clay to the x-ray bombardment and to register the effects of the diffraction caused by the specimen. (See Fig. 4) The goniometer is



NORELCO HIGH ANGLE GONIOMETER
Figure 4

firmly mounted on the same table that supports the x-ray tube and its

shielding. The goniometer itself consists of a frame, drive motor, revolution counter, specimen holder and Geiger-Mueller Tube. This goniometer operates on the principle that a portion of the x-ray beam will be reflected from a sample specimen and will be detected by ionization of the gas inside the Geiger-Mueller Tube. The geometry of this system is shown in Fig. 5. As the specimen is rotated through an angle θ with



GEOMETRY OF THE GONIOMETER
Figure 5

respect to the projected x-ray beam the Geiger-Mueller tube is rotated thru an angle θ with respect to the specimen which means that the G-M tube rotates through angle of 2θ with respect to the projected x-ray beam. It is this 2θ angle that is recorded on the graph paper on the spectrometer and by the revolution counter on the goniometer. The range of the goniometer is from approximately -9° (2θ) to about $+163^\circ$ (2θ). At about $+165^\circ$ (2θ) the Geiger-Mueller tube will hit the top of the x-ray tube housing. To prevent this, micro-switches are installed that will reverse the direction of rotation before any damage can be done. Nevertheless, care should be exercised when operating in this region in case the safety switch should prove to be faulty.

THE X-RAY SPECTROMETER

The x-ray spectrometer is housed in a tall metal cabinet. It consists of several complementary electrical and electronic units that are connected by cables to the Geiger-Mueller tube of the goniometer. The first unit, starting from the bottom and working up, is the amplifier. The next unit is the scaler counter, and the top unit is a Brown Recording Potentiometer. (See Fig. 6) The ionization impulses from the



X-RAY SPECTROMETER

Figure 6

Geiger-Mueller tube are fed through the amplifier and into the scaler counter. The counts per minute indicated by the scaler actuates the recording potentiometer which records the scaler data in the form of a

continuous trace on the roll of chart paper. Simultaneously, the 2θ angle from the goniometer is recorded by means of a second trace on the chart paper.

X-RAY GENERATION FOR FILM WORK

An x-ray tube with a chromium target was used as a source of x-rays. In principle it is similar to the tube diagrammed in Fig. 3. A photograph of this x-ray generating unit is shown in Fig. 7.



Figure 7
X-RAY GENERATING UNIT



Figure 8
DEBYE-SCHERRER CAMERA

X-RAY DIFFRACTION CAMERA

A Debye-Scherrer Powder Camera, with a diameter of 57.3 m.m. was used to produce the strip films used as check data in this work. A photograph of this camera is shown in Fig. 8. This camera operates on

the principle that a portion of the incident x-ray beam will be reflected from a sample specimen and be recorded on a strip of film placed around the inner circumference. The camera, with the specimen in the chuck and the film in place, is adjusted in the path of the x-ray beam. A pinhole system in the camera admits only an extremely thin pencil of x-rays to the camera chamber. This pencil beam must strike the specimen fairly. The adjustment of the specimen in the beam is the most critical and exasperating job in the whole procedure (another vote for the goniometer!). The adjustment of the camera so that the pinhole system lines up with the axis of the beam emitted by the x-ray tube is another painstaking chore. (This adjustment may take as long as one-half hour in itself). The diffracted or reflected beam produces, inside the camera, a cone of radiation. The concentric rings on the film can be considered to be the trace of the cones of radiation on the film. With a perfectly random orientation of crystal planes, a series of perfect and well defined concentric circles will result, forming a pattern that is characteristic of the material being examined. Each ring of the pattern corresponds to one angle of diffraction. Its properties are defined by Bragg's equation which is discussed in the section on theory.

FILM

Eastman Kodak, Type K, Industrial X-Ray Film was used in this work.

DEVELOPING AND PRINTING

Standard Eastman Kodak Co. chemical solutions were used for all developing and printing.

SECTION III

THEORY

GENERAL

In 1912, M. Laue observed that the space lattice of crystalline substances had an interplanar spacing of an order of magnitude of several Angstroms (10^{-8} cm.). He reasoned that x-rays, whose wavelength (λ) is of similar order of magnitude, might be used to study the structure of crystalline materials. His reasoning was based on the fact that the crystal faces would form, in effect, a natural three dimensional ruled grating with just the right distance between rulings to insure diffraction of the x-rays. Subsequent experiments were highly successful. In England, W. L. Bragg and his son (6) were so impressed that they set out to reduce Laue's theory to a more usable form. Their method of analysis has served as the cornerstone for all subsequent diffraction studies. In brief, the Braggs demonstrated that x-ray patterns could be regarded as being produced by mirror like reflections of the electromagnetic waves of the x-ray beam, by regularly spaced plane sheets of atoms which make up the crystal structure (2).

CRYSTAL STRUCTURE

A crystal is essentially a pattern, the atoms being arranged in families or groups in such a manner that the same configuration is repeated at regular intervals in all three dimensions (2). The basic unit, from which the pattern is formed, is called the unit cell. The overall pattern is formed by stacking unit cells one on top of the other. Those surfaces having the highest concentration of atoms or ions are

called crystal faces or planes. In a three dimensional pattern such as is found in crystalline structures, the array of points where the pattern repeats itself is called a space lattice. The actual points themselves are called lattice particles or lattice points. Any plane of a crystal occupied by lattice points is referred to as a crystal or net plane (2). Crystal faces are always parallel to the net planes and furthermore external surfaces are parallel to the densely populated crystal faces.

X-RAYS

X-rays are electromagnetic waves which indicates that they are members of the same spectrum as light waves. The length of a wave caused by an emitter determines its position in this spectrum. Extremely long waves can be felt as heat. As the waves become shorter they reach the visible range. Continuing to even shorter waves, they again become invisible and cannot be felt (2). In this short wave region is a range of wavelengths known as the x-ray zone. The wavelength of these waves is of the order of a few Angstroms ($\text{\AA} = 10^{-8}$ cm.) and they are produced by extremely fast moving electrons impinging on a solid surface. This is the principle of the x-ray tube (see Fig. 3). X-rays upon striking a material cause induced oscillation of the orbital electrons of the atoms they traverse. These electrons radiate energy in all directions when moving toward the nucleus and absorb energy from the x-rays when moving away.

BEHAVIOR OF X-RAYS STRIKING CRYSTAL FACES

In order to obtain diffraction of a wave striking a surface it is necessary to have discontinuities that are of approximately the same

order of magnitude as that of the diffracted wave. Since x-rays have such a short wavelength the discontinuities must be very small. Nature has provided such a diffracting medium in most crystal structures.

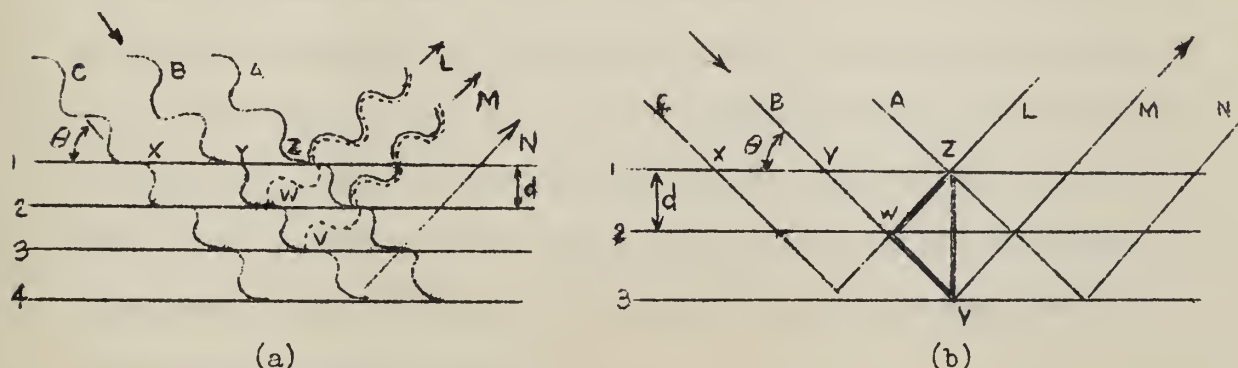


Figure 9
X-RAY BEAM BEING DIFFRACTED THROUGH A CRYSTAL FACE

Fig. 9(a) shows a somewhat simplified version of the behavior of x-rays on striking a crystal. Lines 1,2,3 and 4 represent the lattice planes, at which points the pattern of the arrangement of the atoms or ions is repeated. Let it be assumed that parallel beam of x-rays A, B and C enter the crystal from the left where the wavy lines represent the wavelength of the x-ray beam as well as its direction. Ray A first strikes the crystal plane at point Z and is partially reflected to the right along path L; the remainder of the ray passes through the first plane and is partially reflected by the second plane along path M in a direction parallel to the first reflected ray. It then passes through successive remaining planes as shown, being partially reflected by each plane. To simplify the sketch, the reflections of the two lower rays on striking the first plane at points X and Y have been omitted. That portion of ray B which has not been reflected at point Y will strike

plane 2 at point W and be reflected as shown by the dotted wavy line along path L. Similarly at plane 3, ray B is partially reflected at point V. By the laws of optics, since the wavelength selected is such that the distance between planes corresponds to one-half a wavelength, the partial reflection of wave B from point W reinforces the portion of wave A reflected at point Z. If a wavelength had been selected equal to say one-third of the distance between planes, the various reflected waves would counteract rather than reinforce each other. Therefore, reinforcement is only possible if distance ZW equals one wavelength or some integral multiple thereof.

DERIVATION OF BRAGGS' EQUATION

Referring to Fig. 9(b), it may be said that YW and ZW is equal to $n\lambda$, where n is a positive integer. We can replace the distance ZW by a function of θ (the angle of incidence) and d (the distance between planes). In the triangle ZWV, $\frac{ZW}{ZV} = \sin \theta$; $ZW = n\lambda$ and $ZV = 2d$. Then, from this information we can write the so called Braggs' Law:

$$n\lambda = 2d \sin \theta.$$

This can be readily rearranged to compute the interplanar spacing (d) from x-ray diffraction data, thus:

$$d = \frac{n\lambda}{2 \sin \theta}.$$

POWDER SAMPLE THEORY

The x-ray diffraction theory as originally developed by Laue required the use of well grown and developed crystals, which made the technique of little value to the colloidal physicist. However, P. Debye and P. Scherrer (6) developed a method of x-ray diffraction study for

substances available only in powder form. The reasoning used was that if a crystalline substance is ground to a fine powder, it can be assumed that a mass of this powder will have crystal faces present in absolutely random orientations. Thus if a powder specimen is subjected to an x-ray beam, it is to be expected that the multitude of lattice planes will lie in all of the angles called for by the deflection equation, producing halo or conical pencils of light (2). The position of these cones is recorded on film. Similarly, in the goniometer, it is expected that statistically there will be enough crystal faces in the sample with the proper orientation at any instant to produce a reinforced reflection when the machine attains the proper θ angles.

DATA OBTAINED FROM X-RAY STUDIES

Upon examining x-ray diffraction patterns, it will be observed that the lines have varying intensities depending on the angles at which the reflection occurs, the number and arrangement of the atoms in the lattice, perfection of the crystal irradiated by the x-ray beam and methods employed in recording the diffraction pattern. Actually, the x-ray diffraction pattern represents a summation of all of the components of the crystal. Further, the intensities of the lines revealing variations in arrangement of atoms is an indication of the relative quantities of matter present in the crystal (6). The intensities of lines in film analysis are determined by eye or by means of a densitometer. In x-ray spectrometer analysis the area under the pip is representative of the intensity. It has been found that if a constituent is present to an extent less than ten percent of the sample, the lines representative of

this constituent may be completely obscured by those substances present in larger quantities.

STUDY OF CLAY MINERALS BY X-RAY ANALYSIS (2)

In the application of x-ray diffraction techniques to the identification of clay minerals, the interpretation of the various diffraction patterns requires computation of the interplanar spacing. From the computed spacings, a comparison can be made with some known standard such as Hannawalt's (10) or with results obtained by other investigators from known clay mineral samples. In addition, a comparison of relative intensities of the lines of the individual patterns provides information concerning the amount of the various components making up the clay mineral. Thus, the method of x-ray diffraction has quantitative as well as qualitative aspects.

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SECTION IV

PROCEDURE

GENERAL

Otay, California Montmorillonite (Bentonite); Pioche, Nevada Montmorillonite (Gouge Clay); Jolliet, Illinois Illite; Macon, Georgia Kaolinite and Bedford, Indiana Halloysite were selected as being representative of the overall field of clay minerals. For ionic saturation of these clays it was decided to use Hydrogen, Potassium, Sodium and Calcium since these are of common occurrence in clays in nature and have been used extensively by previous investigators. However, lack of time and non-availability of the machine due to other work required of it by the Metallurgy Dept. forced the investigation to be confined to the first three ionic saturations mentioned.

PARTICLE SIZE

The ~~experiences~~ experiences of previous investigators (2, 3 and 6) have indicated that the sharpest diffraction patterns are obtained with particles passing the 200 mesh sieve. Apparently particles larger than this size give spotty or grainy rings on film and particles passing the 325 mesh screen also give a poor pattern. No attempt was made in this thesis to evaluate the effect of various grain sizes on the spectrometer trace. As a matter of convenience all samples were ground with a mortar and pestle until they passed the 200 mesh sieve, since it was felt that this size would give the best results on the check films. All samples were air dry.

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HOMO-IONIC SATURATION

Peptizing the clay minerals was accomplished by following the procedure adopted by the International Society of Soil Sciences (9). To remove any organic matter that might be present, sixteen grams of the material passing the 200 mesh sieve were boiled for five minutes in thirty cubic centimeters of 6% hydrogen peroxide. At the end of this treatment the mixture was centrifuged and the supernatant liquid poured off. The sample was then treated with twenty cc's of 0.17 N hydrochloric acid in order to remove any unknown ions that might be absorbed on the particles in the as received condition. After stirring the HCl and clay mixture for two minutes the mixture was centrifuged and the supernatant decanted. This yielded an H^+ saturated clay, which was then washed three times with distilled water to remove the dissolved carbonates and replaced ions.

The sample was then divided into four equal parts. One part was marked H^+ Saturated mineral and placed in the oven at $105^\circ C$. The remaining three batches were each mixed with an 0.008 N solution of potassium, sodium and calcium hydroxide respectively. Each mixture was beaten in a high speed stirrer for twenty minutes. After this it was centrifuged for approximately ten minutes and the supernatant removed. These peptized clay batches were each placed in a small beaker, marked and placed in an oven at $105^\circ C$ for twenty-four hours. After the saturated clays were dry, they were again ground with a mortar and pestle so that they passed a 200 mesh sieve. They were then stored in test tubes exposed to the air until used, no attempt being made to maintain an oven

The first of the year was a very successful one for the
company. The sales were up to the mark and the
profits were also very good. The management
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the future prospects were very bright.
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dry condition. Each of the five clays was given a homo-ionic saturation with each of the ions H^+ , K^+ , Na^+ and Ca^{++} .

PROCESSING SAMPLE FOR GONIOMETER

The first probelm was fabrication of four sample holders for the goniometer. The holders are available commercially but were considered too expensive. The holders were made from aluminum 1/16" thick and conforming to the dimensions shown in Fig. 10.

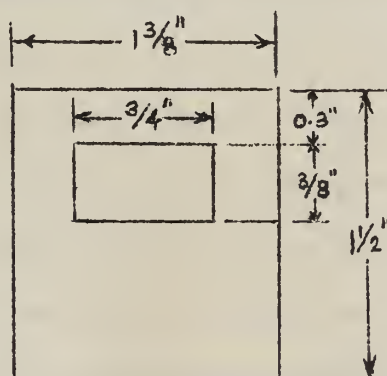


Figure 10
SPECIMEN HOLDER FOR THE GONIOMETER

Sufficient material (approximately two grams) to fill the opening in the sample holder is thoroughly mixed with a suitable binder. The sample holder is laid flat on a piece of waxed paper and the mixture of clay and binder is buttered into the opening with a spatula and struck off flush. The technique is easily acquired after a few trials.

Previous investigators (2 and 3) used Duco Household Cement and Collodion as binders in preparing samples for extrusion. Both materials were tried for this thesis and Duco Cement was finally settled upon as the better material since it does not shrink and crack on drying as much as the collodion. It was not considered necessary to dry the samples in



the oven since both binders are relatively rapid in setting up. It was soon found that it was better to leave the sample heaped up above the face of the holder since the sample shrank down flush on drying. When the extra thickness of sample was not supplied, the dry sample shrank away from the sides of the opening and sank below the face of the holder in the middle.

PROCESSING SAMPLE FOR PHOTOGRAPHING WITH X-RAYS

A glass rod was heated until plastic and pulled to neck it down and furnish a fine rod of small diameter. When cool this rod was broken loose and coated with stopcock grease. The greased rod was then rolled in a small quantity of clay mineral passing the 200 mesh sieve. This put a powder coating on the glass rod which was then mounted in the chuck of the Debye-Scherrer camera and exposed to the x-rays. Another easy method for preparing the powder sample is the extrusion process (2 and 3). Both methods give good results. See Appendix C for check films.

PROCEDURE FOR EXPOSING GONIOMETER SPECIMEN

The specimen holder with the powder specimen in place was inserted in the clip holder on the goniometer, shields adjusted and a 1° slit placed over the port of the x-ray tube. The drive gears were set to give 2° of 2θ rotation per minute and then the spectrometer and the x-ray tube were turned on and allowed to warm up for five minutes. The goniometer was always started from the $+4^\circ$ 2θ position and allowed to travel in an increasing 2θ direction. As soon as the equipment had

The subject of this volume is the history of the American people from the first settlement of the continent to the present time. It is a history of the growth of the nation, of the development of its institutions, and of the progress of its civilization. It is a history of the struggles and triumphs of a people who have built a great and glorious nation out of a wilderness of ignorance and poverty.

THE AMERICAN PEOPLE

The first chapter of this volume is devoted to the history of the American people from the first settlement of the continent to the present time. It is a history of the growth of the nation, of the development of its institutions, and of the progress of its civilization. It is a history of the struggles and triumphs of a people who have built a great and glorious nation out of a wilderness of ignorance and poverty. The second chapter is devoted to the history of the American people from the first settlement of the continent to the present time. It is a history of the growth of the nation, of the development of its institutions, and of the progress of its civilization. It is a history of the struggles and triumphs of a people who have built a great and glorious nation out of a wilderness of ignorance and poverty.

THE AMERICAN PEOPLE

The third chapter of this volume is devoted to the history of the American people from the first settlement of the continent to the present time. It is a history of the growth of the nation, of the development of its institutions, and of the progress of its civilization. It is a history of the struggles and triumphs of a people who have built a great and glorious nation out of a wilderness of ignorance and poverty. The fourth chapter is devoted to the history of the American people from the first settlement of the continent to the present time. It is a history of the growth of the nation, of the development of its institutions, and of the progress of its civilization. It is a history of the struggles and triumphs of a people who have built a great and glorious nation out of a wilderness of ignorance and poverty.

warmed up sufficiently a zero check was made of the meter on the spectrometer and the control dials were placed on the settings shown in Fig. 11.

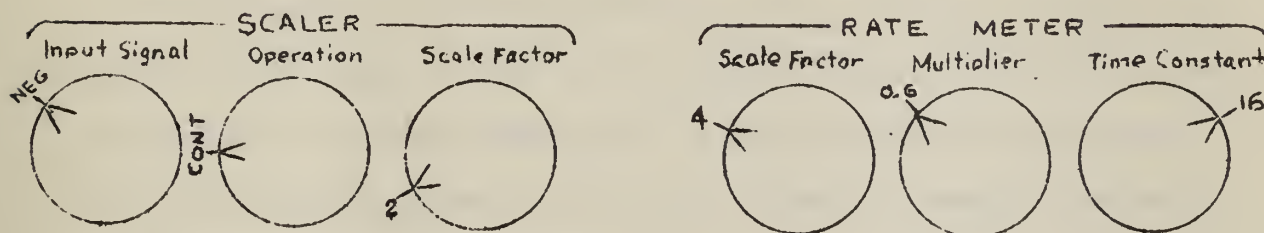


Figure 11
DIAL SETTINGS FOR THE SCALER COUNTER

With these preliminaries out of the way the port cover on the tube was opened, the clutch engaged on the drive mechanism of the goniometer and the drive turned ON on the recording potentiometer. At $80^\circ 2\theta$ the drives were turned off while a 4° slit was substituted for the 1° slit in front of the tube port. The drives were then turned on and the trace recording continued. The charts obtained from this machine are shown in Appendix B. Five films were run of samples selected at random from the homo-conic materials to serve as a check on the accuracy of results obtained with the x-ray spectrometer. (See Appendix C)

READING THE SPECTROMETER CHART AND COMPUTING THE INTERPLANAR SPACING

The spectrometer charts (see Appendix B) are extremely easy to read. The intensity of the pips was determined by computing the area under them to any arbitrary scale. In this case the base and height of the pip was measured with the #10 scale on an engineer's scale and the area computed as a simple triangle. (See columns labeled "I" in Appendix A) It would be just as easy to planimeter the pips and use the planimeter reading direct, as the intensity value. The system doesn't matter as

long as it is applied consistently, since we are interested only in relative intensity.

The 2θ angle was ascertained by placing a triangle or other transparent straight edge through the high point of the pip, parallel to the vertical grid lines and reading the 2θ angle directly on the degrees 2θ trace along the bottom of the chart. From this reading, the value of $2\sin\theta$ is easily calculated.

The value of $n\lambda$ is known from the tube characteristics. For a chromium target tube it is 2.28962 \AA . For slide rule calculation, the value 2.29 \AA was used as being accurate enough.

Knowing the values of $n\lambda$ and $2\sin\theta$ it is only one operation on a slide rule to determine the interplanar spacing (d) by means of the formula:

$$d = \frac{n\lambda}{2\sin\theta}.$$

READING THE FILM DIFFRACTION PATTERN AND COMPUTING THE INTERPLANAR SPACING

The radii of all visible rings on the films were read by placing the film over a light source and measuring the distance from the center of the beam in centimeters. This distance is designated "s". The distance from the specimen to the film inside the camera is equal to $1/2$ the diameter of the camera and is designated "r". Then $\frac{s}{r} = 2\theta$ (radians) and $\theta = (\frac{s}{r})(\frac{90}{\pi})$. Again $r\lambda$ is known from the tube characteristics. The Bragg's equation is used in the form shown below for the film work:

$$d = \frac{n\lambda}{2\sin(\frac{90}{\pi} \frac{s}{r})}$$

1911

The following is a list of the names of the persons who have been elected to the office of the President of the United States since the year 1789. The names are given in alphabetical order, and the year of election is given in parentheses. The names are given in the order in which they were elected, and the year of election is given in parentheses. The names are given in the order in which they were elected, and the year of election is given in parentheses.

George Washington (1789)
John Adams (1797)
Thomas Jefferson (1801)
James Madison (1809)
James Monroe (1817)
John Quincy Adams (1825)
Andrew Jackson (1829)
Martin Van Buren (1837)
William Henry Harrison (1841)
Francis Pickens (1858)
Abraham Lincoln (1861)
Andrew Johnson (1865)
Ulysses S. Grant (1869)
Rutherford B. Hayes (1877)
James A. Garfield (1881)
Chester A. Arthur (1881)
Grover Cleveland (1895)
William McKinley (1897)
Theodore Roosevelt (1901)
Woodrow Wilson (1913)
Warren G. Harding (1921)
Calvin Coolidge (1923)
Herbert Hoover (1929)
Franklin D. Roosevelt (1933)
Dwight D. Eisenhower (1953)
John F. Kennedy (1961)
Lyndon B. Johnson (1963)
Richard M. Nixon (1969)
Jimmy Carter (1977)
Ronald Reagan (1981)
George H. W. Bush (1989)
Bill Clinton (1993)
George W. Bush (2001)
Barack Obama (2009)
Donald Trump (2017)

where s and r are in cm., $n\lambda$ is in \AA and d is in \AA .

INTERPRETATION OF DIFFRACTION PATTERNS

The interplanar spacings together with the relative intensities of the lines give a "fingerprint" of the particular clay mineral being investigated. By comparing the pattern of an unknown clay with the patterns for known clay minerals an identification of the unknown material can be made.

The Hannawalt Method (10) is based on this fact but uses only the three most intense lines. The data for the three intense lines is published in sets of index cards put out by the A.S.T.M. A comparison between the results of the samples run in this thesis is made in Appendix D.

THE UNIVERSITY OF CHICAGO

DEPARTMENT OF CHEMISTRY

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SECTION V

RESULTS AND CONCLUSIONS

GENERAL

The results of the tests run are tabulated in the various appendices to this thesis. Of particular interest is Appendix D where a comparison is made between the goniometer - spectrometer results and the interplanar spacings listed by the A.S.T.M. as standard for these clay minerals. There is no indication on the A.S.T.M. cards as to what ion is predominant on the clay mineral listed and in the absence of any definite information on this point it must be presumed that the clays in question were tested in an air-dry, as received, condition. This supposition seems to be born out by the results achieved by Cline and Hediger⁽²⁾, who investigated their clays in the oven-dry and fully hydrated states. The A.S.T.M. values seem to fall generally between the extreme values obtained by the aforementioned investigators.

The results obtained by using the K^+ ion to saturate the clay minerals seem to be consistently the best match with A.S.T.M. standards, with H^+ saturated material running a close second. Since saturation with the H^+ ion is the easier situation to achieve, it is concluded that H^+ saturated clay is the one to use.

The discrepancies between the results obtained and the standard A.S.T.M. values are considered to be due to three major factors, namely:

- (a) Differences in sample preparation technique. This can definitely affect the resulting diffraction pattern, especially in the

case of those clay minerals having an expanding lattice type structure such as the Montmorillonites. With this structure the interplanar spacing is different for each different adsorbed ion (9). Variations in grain size will also affect the diffraction pattern and so will the moisture content.

(b) Spill over of the divergent x-ray beam at very low angles.

From the geometry of the goniometer (see Fig. 5) it will be noticed that at low angles the sample holder and Geiger-Mueller tube are practically in line with the tube port and are both in the direct path of the x-ray beam. This peculiarity of the goniometer gives rise to speculation as to just how effective the machine would be in analyzing certain fully saturated Montmorillonites. A possible solution to this difficulty will be discussed under recommendations. (Section VI)

(c) Differences in resolution ability of the two methods. It is considered very difficult to resolve visually two bands that are of fairly strong intensity and that occur close together on the film. The literature is full of remarks to this effect. Even the A.S.T.M. cards contain comments such as "Diffuse Pattern", etc. On the spectrometer trace, although the peaks often have broad tops, there is usually a very good indication when two peaks are trying to overlap.

Schieltz (12) has stated that, for purposes of comparing diffraction patterns in identification work, an agreement within $\pm 0.05 \text{ \AA}$ in the

computed interplanar spacings should be considered accurate. The agreement between the spectrometer traces and the A.S.T.M. standards was well within this tolerance, with but a few exceptions. These exceptions can be easily explained by the causes for discrepancies listed above. The differences between the orders of intensity listed by the A.S.T.M. and those recorded by the spectrometer are not so easily explained. In some cases it would seem that it is a question of technique in interpreting and computing intensity from the spectrometer trace. In other cases the discrepancy is too great for this explanation and we can take our choice between doubting the results given on the A.S.T.M. cards and questioning the sample preparation technique and operating technique for the spectrometer.

EVALUATION OF THE GONIOMETER - SPECTROMETER

The results of this limited series of tests indicate that the goniometer - spectrometer is an excellent tool for the identification of clay minerals by x-ray diffraction. The time required for sample preparation is no longer than that required for film diffraction. The exposure time is about one-half that required in film work. No developing or drying is required. If, for any reason, the x-ray tube shuts off it is known immediately rather than having to waste time developing a blank film. The recorded diffraction pattern is easier to read and the required computations are simpler than for the film record. Finally the computed interplanar spacings are closer to A.S.T.M. standards than those obtained by any other recent investigators (2, 3) at R.P.I., all of whom worked with film.

Since this very limited test has shown such highly gratifying results it may be expected that further investigation and experimentation with such a flexible and versatile machine will eliminate many of the discrepancies that showed up in this first trial. (See recommendations Section VI)

EVALUATION OF SAMPLE PREPARATION TECHNIQUE

The technique used in preparing the powder samples for this thesis is described in Section IV and was based on work by Dearth and Williams⁽³⁾ as refined by Cline and Hediger⁽²⁾. It is felt that this technique is adequate and it is believed that for simple identification work no further modification is needed at this time. However, from a thorough investigation standpoint, it is considered adviseable to check the effects of both larger and smaller grain sizes on the character of the diffraction pattern produced by the goniometer - spectrometer. Duco cement is considered to be the best binder for powder samples to be used in this machine.

CONCLUSIONS

The goniometer - spectrometer a superior analytical tool and should be investigated further to insure that optimum advantage is being taken of its flexibility. A series of further tests is outlined in Section VI.

SECTION VI

RECOMMENDATIONS

From the results and conclusions obtained in this thesis work certain recommendations suggested themselves. These recommendations are combined in this section under the broad headings of further research, classes and laboratory, procurement, and methods. They are not intended to be critical of department policy or of individuals. They are set down in the hope that they may be of some use to those who follow the author as students in this institution and to those responsible for the guidance and training of the students. With this thought in mind the following recommendations are respectfully offered.

FURTHER RESEARCH

Before a truly definitive evaluation can be made of the goniometer - spectrometer for clay mineral analysis, certain minimum tests should be run. These tests are more than can be accomplished in one or two semesters by students who must carry a full academic load in addition to their thesis work. Therefore the obvious tests are set forth in increments, each increment of which is recommended as a separate thesis. Each increment will not only investigate a distinct phase of clay mineral analysis but will add more traces to a sort of "master finger print" file that will eventually aid materially in rapid identification of unknown samples

1. Individual pure known clays saturated with combinations of two or more known ions. (Air dry)

CHAPTER IV

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CHAPTER V

THEORY OF THE

CHAPTER VI

THEORY OF THE

2. Known mixtures of pure known clays saturated with homo-ionic saturation. (Air dry)
3. Known mixtures of pure known clays saturated with mixtures of two or more known ions. (Air dry)
4. Pure known clays in an as recieved condition (i.e. with unknown ions adsorbed on them).
5. Effect of grain size on the trace and the effect of a small amount of sand mixed with the clay. (Try - pass 140, ret. 200 - 325 to pan - etc.)
6. If necessary, run oven dry and fully hydrated tests on the above samples (only necessary if the air dry samples do not give useably consistent results)
7. Adjust size of opening in sample holder; try sample holders of plexiglas, bakelite and other amorphous substances; study shape of pips on the trace to determine if the shape is indicative of the moisture content and/or the adsorbed ion (this study will require a large backlog of spectrometer charts).
8. Design, manufacture and test a new slit system for the goniometer that will give discrimination at low angles (Dr. A.A. Burr of the Metallurgy Department has some ideas on this subject that are very pertinent).
9. Run tests on unknown clay minerals (with all the above background to refer to, identification of unknowns should be feasible)

CLASSES AND LABORATORY

It is recommended that the laboratory curriculum in soil mechanics be revised to include at least one experiment or demonstration in x-ray analysis.

It is further recommended that H.C. Schieltz' excellent article (12) on "X-Ray Analysis" be included as required reading in the Soil Mechanics III course.

PROCUREMENT

It is strongly recommended that every effort be made to obtain a goniometer - spectrometer for the Soil Mechanics Division. It is realized that the equipment in question is expensive and the recommendation is not made lightly. Even after a relatively superficial evaluation this type of equipment appears so highly superior to the film process that the extra expense is considered well worth while.

The difference in cost between the two systems is not as great as it would first appear when it is considered that a complete darkroom with all its equipment must be included in the initial cost for the film process.

The procurement of this x-ray equipment would provide facilities for laboratory exercises and would eliminate the fight for time that arises in trying to do thesis work on the Metallurgy Department's machine. Interference was encountered during the course of this thesis work from laboratory sections interfering and from members of the Metallurgy Department who required the machine for their own thesis work.

THE HISTORY OF THE

REIGN OF KING CHARLES THE FIRST

IN THE YEAR 1649

BY JOHN BURNET

OF THE UNIVERSITY OF OXFORD

IN TWO VOLUMES

LONDON: Printed by J. Streater, at the Sign of the Gun, in St. Dunstons Church-yard, 1680.

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METHODS

It is recommended that in preparing powder specimens for future research work, of a nature similar to this thesis, the processes developed by previous investigators (2, 3) be continued in use.

It is recommended that the procedure of the International Society of Soil Science⁽⁹⁾ for peptizing clay minerals be continued in use.

APPENDIX A

COMPUTED INTERPLANAR SPACINGS H^+ Saturated Montmorillonite (Bentonite)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
108	8.6°	4.3°	0.075	0.150	2.29	15.25
8	62.0°	31.0°	0.515	1.030	"	2.22
18	69.0°	34.5°	0.566	1.132	"	2.02
42	99.6°	49.8°	0.764	1.528	"	1.50
65	107.0°	53.5°	0.804	1.608	"	1.43
16	112.8°	56.4°	0.833	1.666	"	1.38
15	117.4°	58.7°	0.854	1.708	"	1.34
90	139.8°	69.9°	0.939	1.878	"	1.22

 K^+ Saturated Montmorillonite (Bentonite)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
280	8.6°	4.3°	0.075	0.150	2.29	15.25
18	20.5°	10.2°	0.178	0.356	"	6.48
10	27.4°	13.7°	0.237	0.474	"	4.84
25	30.0°	15.0°	0.259	0.518	"	4.42
16	53.6°	26.8°	0.451	0.902	"	2.54
6	58.8°	29.4°	0.491	0.982	"	2.33
3	62.2°	31.1°	0.517	1.034	"	2.21
2	69.0°	34.5°	0.566	1.132	"	2.02
36	99.6°	49.8°	0.764	1.528	"	1.50
14	107.0°	53.5°	0.804	1.608	"	1.43
6	111.0°	55.5°	0.824	1.648	"	1.39
9	112.8°	56.4°	0.833	1.666	"	1.38
12	117.0°	58.5°	0.853	1.706	"	1.34
3	119.4°	59.7°	0.863	1.726	"	1.33
25	125.0°	62.5°	0.887	1.774	"	1.29
5	128.8°	64.4°	0.902	1.804	"	1.27
15	133.8°	66.9°	0.920	1.840	"	1.25
10	136.2°	68.6°	0.931	1.862	"	1.23
28	139.8°	69.9°	0.939	1.878	"	1.22

18

18

Na⁺ Saturated Montmorillonite (Bentonite)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
188	8.6°	4.3°	0.075	0.150	2.29	15.25
2	14.2°	7.1°	0.124	0.248	"	9.24
5	26.2°	13.1°	0.227	0.454	"	5.05
4	27.4°	13.7°	0.237	0.474	"	4.84
28	30.0°	15.0°	0.259	0.518	"	4.42
22	53.6°	26.8°	0.451	0.902	"	2.54
13	58.6°	29.3°	0.489	0.978	"	2.34
4	62.2°	31.1°	0.517	1.034	"	2.21
18	69.0°	34.5°	0.566	1.132	"	2.02
10	93.6°	46.8°	0.729	1.458	"	1.57
30	99.6°	49.8°	0.764	1.528	"	1.50
27	106.4°	53.2°	0.801	1.602	"	1.43
8	117.4°	58.7°	0.854	1.708	"	1.34
36	125.2°	62.6°	0.888	1.776	"	1.29
84	139.8°	69.9°	0.939	1.878	"	1.22

H⁺ Saturated Montmorillonite (Gouge Clay)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
59	8.8°	4.4°	0.077	0.154	2.29	14.89
8	19.0°	9.5°	0.165	0.330	"	6.94
6	29.8°	14.9°	0.257	0.514	"	4.47
20	44.5°	22.2°	0.379	0.758	"	3.03
8	53.2°	26.6°	0.448	0.896	"	2.56
25	58.6°	29.3°	0.489	0.978	"	2.34
10	62.2°	31.1°	0.517	1.034	"	2.22
32	69.2°	34.6°	0.568	1.136	"	2.02
30	96.4°	48.2°	0.745	1.490	"	1.54
35	100.4°	50.2°	0.768	1.536	"	1.49
42	106.5°	53.2°	0.801	1.602	"	1.43
7	113.4°	56.7°	0.836	1.672	"	1.37
12	117.3°	58.65°	0.854	1.708	"	1.34
12	124.2°	62.1°	0.884	1.768	"	1.30
9	133.5°	66.7°	0.919	1.838	"	1.25
122	139.8°	69.9°	0.939	1.878	"	1.22
10	146.6°	73.3°	0.958	1.916	"	1.19

K⁺ Saturated Montmorillonite (Gouge Clay)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	n λ	d(Å)
90	8.8°	4.4°	0.077	0.154	2.29	14.89
32	30.2°	15.1°	0.261	0.522	"	4.39
6		19.0°	0.326	0.652	"	3.52
14	45.4°	22.7°	0.386	0.772	"	2.96
7	50.2°	25.1°	0.424	0.848	"	2.70
7	53.2°	26.6°	0.448	0.896	"	2.56
10	58.6°	29.3°	0.489	0.978	"	2.34
10	62.4°	31.2°	0.518	1.036	"	2.21
8	69.0°	34.5°	0.566	1.132	"	2.02
12	86.3°	43.15°	0.684	1.368	"	1.67
30	99.6°	49.8°	0.764	1.528	"	1.50
6	106.5°	53.25°	0.801	1.602	"	1.43
12	117.4°	58.7°	0.854	1.708	"	1.34
10	124.0°	62.0°	0.883	1.766	"	1.30
49	139.6°	69.8°	0.939	1.878	"	1.22

Na⁺ Saturated Montmorillonite (Gouge Clay)

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	n λ	d(Å)
116	8.8°	4.4°	0.077	0.154	2.29	14.89
12	18.4°	9.2°	0.160	0.320	"	7.16
20	29.6°	14.8°	0.255	0.510	"	4.50
10	37.5°	18.7°	0.321	0.642	"	3.57
18	44.2°	22.1°	0.376	0.752	"	3.05
8	53.0°	26.5°	0.446	0.892	"	2.57
27	58.7°	29.3°	0.489	0.978	"	2.34
15	62.2°	31.1°	0.517	1.034	"	2.22
20	69.0°	34.5°	0.566	1.132	"	2.02
6	89.8°	44.9°	0.705	1.410	"	1.62
5	93.5°	46.7°	0.728	1.456	"	1.57
10	99.6°	49.8°	0.764	1.528	"	1.50
8	106.6°	53.3°	0.802	1.604	"	1.43
9	117.3°	58.6°	0.854	1.708	"	1.34
6	129.6°	64.8°	0.905	1.810	"	1.27
11	133.4°	66.7°	0.918	1.836	"	1.25
5	136.4°	68.2°	0.928	1.856	"	1.23
105	139.8°	69.9°	0.939	1.878	"	1.22
45	157.2°	78.6°	0.981	1.962	"	1.17

H^+ Saturated Illite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
118	12.0°	6.0°	0.105	0.210	2.29	10.91
13	16.8°	8.4°	0.146	0.292	"	7.85
17	26.8°	13.4°	0.232	0.464	"	4.95
28	31.8°	15.9°	0.274	0.548	"	4.18
14	36.5°	18.2°	0.312	0.624	"	3.68
90	40.2°	20.1°	0.344	0.688	"	3.34
10	53.2°	26.6°	0.448	0.896	"	2.56
14	58.8°	29.4°	0.491	0.982	"	2.34
12	68.8°	34.4°	0.565	1.130	"	2.03
40	87.8°	43.9°	0.693	1.386	"	1.65
16	96.2°	48.1°	0.744	1.488	"	1.54
21	99.5°	49.8°	0.764	1.528	"	1.50
24	106.4°	53.2°	0.801	1.602	"	1.43
65	113.2°	56.6°	0.835	1.670	"	1.37
35	117.4°	58.7°	0.854	1.708	"	1.34
70	124.6°	62.3°	0.885	1.770	"	1.29
24	132.6°	66.3°	0.916	1.832	"	1.25
75	139.8°	69.9°	0.939	1.878	"	1.22
25	146.0°	73.0°	0.956	1.912	"	1.20
56	151.8°	75.9°	0.970	1.940	"	1.18

 K^+ Saturated Illite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
24	12.2°	6.1°	0.106	0.212	2.29	10.80
6	17.2°	8.6°	0.150	0.300	"	7.54
7	19.8°	9.9°	0.172	0.344	"	6.76
9	26.6°	13.3°	0.230	0.460	"	4.98
21	36.4°	18.2°	0.312	0.624	"	3.68
42	40.2°	20.1°	0.344	0.688	"	3.34
20	53.0°	26.5°	0.446	0.892	"	2.57
6	58.8°	29.4°	0.491	0.982	"	2.34
10	69.0°	34.5°	0.566	1.132	"	2.02
18	87.8°	43.9°	0.693	1.386	"	1.65
17	96.0°	48.0°	0.743	1.486	"	1.54
24	99.4°	49.7°	0.763	1.526	"	1.50
32	106.5°	53.2°	0.801	1.602	"	1.43
28	113.2°	56.6°	0.835	1.670	"	1.37
7	117.4°	58.7°	0.854	1.708	"	1.34
19	125.0°	62.5°	0.887	1.774	"	1.29
12	131.6°	65.8°	0.912	1.824	"	1.26
90	139.8°	69.9°	0.939	1.878	"	1.22
24	145.6°	72.8°	0.955	1.910	"	1.20
50	151.6°	75.8°	0.969	1.938	"	1.18

Na⁺ Saturated Illite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
102	12.0°	6.0°	0.105	0.210	2.29	10.91
6	17.6°	8.8°	0.153	0.306	"	7.49
8	19.6°	9.3°	0.162	0.324	"	7.07
18	26.6°	13.3°	0.230	0.460	"	4.98
8	29.8°	14.9°	0.257	0.514	"	4.46
4	31.2°	15.6°	0.269	0.538	"	4.26
9	34.2°	17.1°	0.294	0.588	"	3.89
15	36.3°	18.2°	0.312	0.624	"	3.68
90	40.2°	20.1°	0.344	0.688	"	3.34
17	53.0°	26.5°	0.446	0.892	"	2.57
4	55.6°	27.8°	0.466	0.932	"	2.46
10	58.8°	29.4°	0.491	0.982	"	2.34
15	62.2°	31.1°	0.517	1.034	"	2.21
9	65.0°	32.5°	0.537	1.074	"	2.13
15	68.8°	34.4°	0.565	1.130	"	2.03
14	78.0°	39.0°	0.629	1.258	"	1.82
65	88.2°	44.1°	0.696	1.392	"	1.65
18	96.2°	48.1°	0.744	1.488	"	1.54
50	99.6°	49.8°	0.764	1.528	"	1.50
7	104.2°	52.1°	0.789	1.578	"	1.45
15	106.6°	53.3°	0.802	1.604	"	1.43
78	113.2°	56.6°	0.835	1.670	"	1.37
16	117.4°	58.7°	0.854	1.708	"	1.34
75	125.4°	62.7°	0.889	1.778	"	1.29
20	132.5°	66.7°	0.918	1.836	"	1.25
100	139.6°	69.8°	0.939	1.878	"	1.22
42	145.8°	72.9°	0.956	1.912	"	1.20
110	152.0°	76.0°	0.970	1.940	"	1.18
18	157.2°	78.6°	0.980	1.960	"	1.17

H⁺ Saturated Kaolinite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
330	18.5°	9.2°	0.161	0.322	2.29	7.12
38	34.2°	17.1°	0.294	0.588	"	3.90
190	37.5°	18.7°	0.321	0.642	"	3.57
61	58.6°	29.3°	0.489	0.978	"	2.34
36	69.0°	34.5°	0.566	1.132	"	2.02
59	87.4°	43.7°	0.691	1.382	"	1.66
59	106.4°	53.2°	0.801	1.602	"	1.43
45	117.6°	58.8°	0.855	1.710	"	1.34
36	123.0°	61.5°	0.879	1.758	"	1.30
24	126.5°	63.2°	0.893	1.786	"	1.28
130	139.6°	69.8°	0.939	1.878	"	1.22

K^+ Saturated Kaolinite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
469	18.2°	9.1°	0.158	0.316	2.29	7.25
48	33.8°	16.9°	0.291	0.582	"	3.94
161	37.2°	18.6°	0.319	0.638	"	3.59
36	53.2°	26.6°	0.448	0.896	"	2.55
35	58.8°	29.4°	0.491	0.982	"	2.33
16	69.0°	34.5°	0.566	1.132	"	2.02
72	86.8°	43.4°	0.687	1.374	"	1.67
28	100.6°	50.3°	0.769	1.538	"	1.49
32	107.0°	53.5°	0.804	1.608	"	1.43
28	117.6°	58.8°	0.855	1.710	"	1.34
32	122.6°	61.3°	0.877	1.754	"	1.31
18	126.4°	63.2°	0.893	1.786	"	1.28
48	139.6°	69.8°	0.939	1.878	"	1.22

 Na^+ Saturated Kaolinite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
778	18.4°	9.2°	0.160	0.320	2.29	7.16
96	33.6°	16.8°	0.289	0.578	"	3.96
414	37.2°	18.6°	0.319	0.638	"	3.59
528	58.3°	29.15°	0.487	0.974	"	2.36
48	87.2°	43.6°	0.690	1.380	"	1.66
18	96.4°	48.2°	0.745	1.490	"	1.54
15	100.7°	50.35°	0.770	1.540	"	1.49
33	107.0°	53.5°	0.804	1.608	"	1.43
24	117.5°	58.75°	0.855	1.710	"	1.34
55	122.6°	61.3°	0.877	1.754	"	1.31
60	139.8°	69.9°	0.939	1.878	"	1.22
72	148.0°	74.0°	0.961	1.922	"	1.19

H^+ Saturated Halloysite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
75	18.2°	9.1°	0.158	0.316	2.29	7.25
5	20.8°	10.4°	0.181	0.362	"	6.34
3	25.4°	12.7°	0.220	0.440	"	5.20
18	27.3°	13.6°	0.235	0.470	"	4.87
54	30.2°	15.1°	0.261	0.522	"	4.39
16	36.6°	18.3°	0.314	0.628	"	3.65
6	39.6°	19.8°	0.339	0.678	"	3.38
25	48.6°	24.3°	0.412	0.824	"	2.78
20	58.6°	29.3°	0.489	0.978	"	2.34
5	62.0°	31.0°	0.515	1.030	"	2.22
14	69.0°	34.5°	0.566	1.132	"	2.02
12	93.4°	46.7°	0.728	1.456	"	1.57
66	101.2°	50.6°	0.773	1.546	"	1.48
18	106.4°	53.2°	0.801	1.602	"	1.43
13	117.4°	58.7°	0.854	1.708	"	1.34
10	127.2°	63.6°	0.896	1.792	"	1.28
9	137.0°	68.5°	0.930	1.860	"	1.23
113	139.7°	69.9°	0.939	1.878	"	1.22
35	157.6°	78.8°	0.981	1.962	"	1.17

 K^+ Saturated Halloysite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
85	18.2°	9.1°	0.158	0.316	2.29	7.25
15	27.4°	13.7°	0.236	0.472	"	4.85
32	30.4°	15.2°	0.262	0.524	"	4.38
3	34.4°	17.2°	0.296	0.592	"	3.87
35	36.8°	18.4°	0.315	0.630	"	3.64
16	53.6°	26.8°	0.451	0.902	"	2.54
8	58.6°	29.3°	0.489	0.978	"	2.34
3	62.2°	31.1°	0.516	1.032	"	2.22
12	69.0°	34.5°	0.566	1.132	"	2.02
18	89.2°	44.6°	0.702	1.404	"	1.63
64	101.4°	50.8°	0.775	1.550	"	1.48
15	106.4°	53.2°	0.801	1.602	"	1.43
7	113.5°	56.7°	0.836	1.672	"	1.37
12	126.6°	63.3°	0.893	1.786	"	1.28
56	139.7°	69.9°	0.939	1.878	"	1.22
8	153.2°	76.6°	0.973	1.946	"	1.18
14	156.6°	78.3°	0.979	1.958	"	1.17

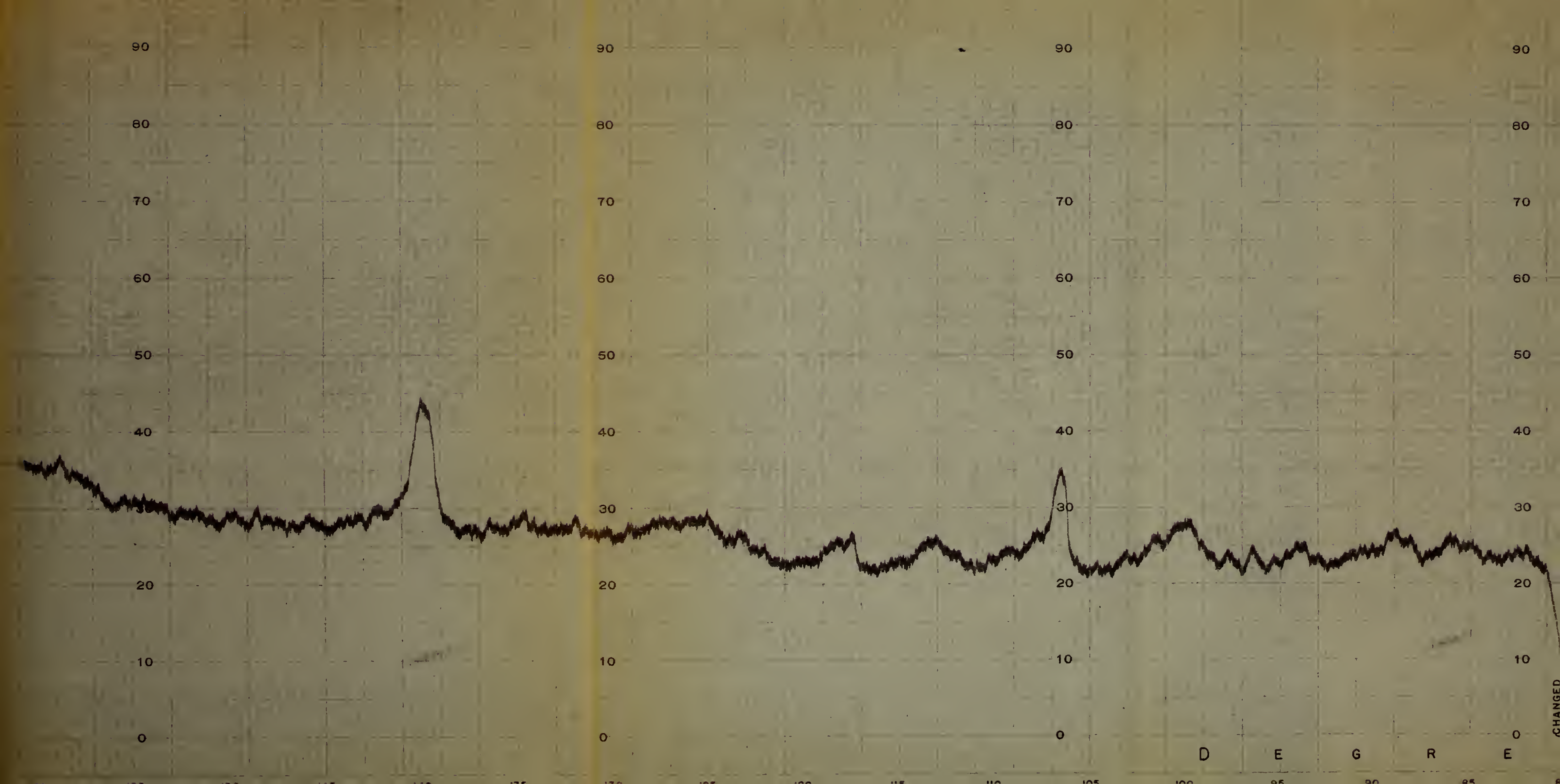
Na⁺ Saturated Halloysite

I	2 θ	θ	$\sin \theta$	2 $\sin \theta$	$n\lambda$	$d(\text{\AA})$
85	18.2°	9.1°	0.158	0.316	2.29	7.25
10	27.2°	13.6°	0.235	0.470	"	4.87
28	30.4°	15.2°	0.262	0.524	"	4.38
20	37.2°	18.6°	0.319	0.638	"	3.59
5	53.6°	26.8°	0.451	0.902	"	2.54
14	58.6°	29.3°	0.489	0.978	"	2.34
3	62.0°	31.0°	0.515	1.030	"	2.22
4	69.0°	34.5°	0.566	1.132	"	2.02
49	101.4°	50.8°	0.775	1.550	"	1.48
21	106.4°	53.2°	0.801	1.602	"	1.43
10	109.6°	54.8°	0.817	1.634	"	1.40
8	117.4°	58.7°	0.854	1.708	"	1.34
3	126.6°	63.3°	0.893	1.786	"	1.28
20	136.5°	68.2°	0.928	1.856	"	1.23
70	139.8°	69.9°	0.939	1.878	"	1.22
30	158.2°	79.1	0.982	1.964	"	1.17

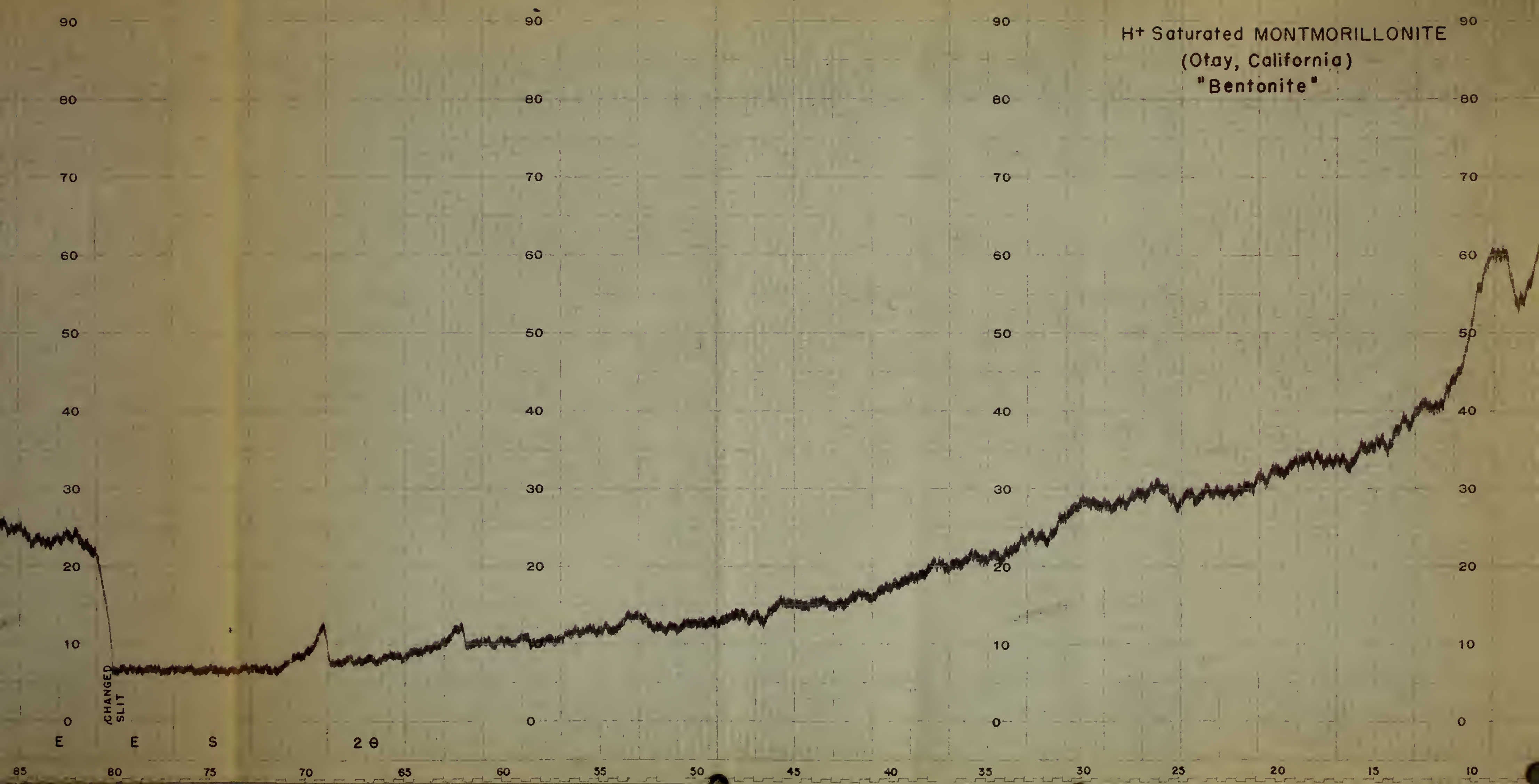
APPENDIX B

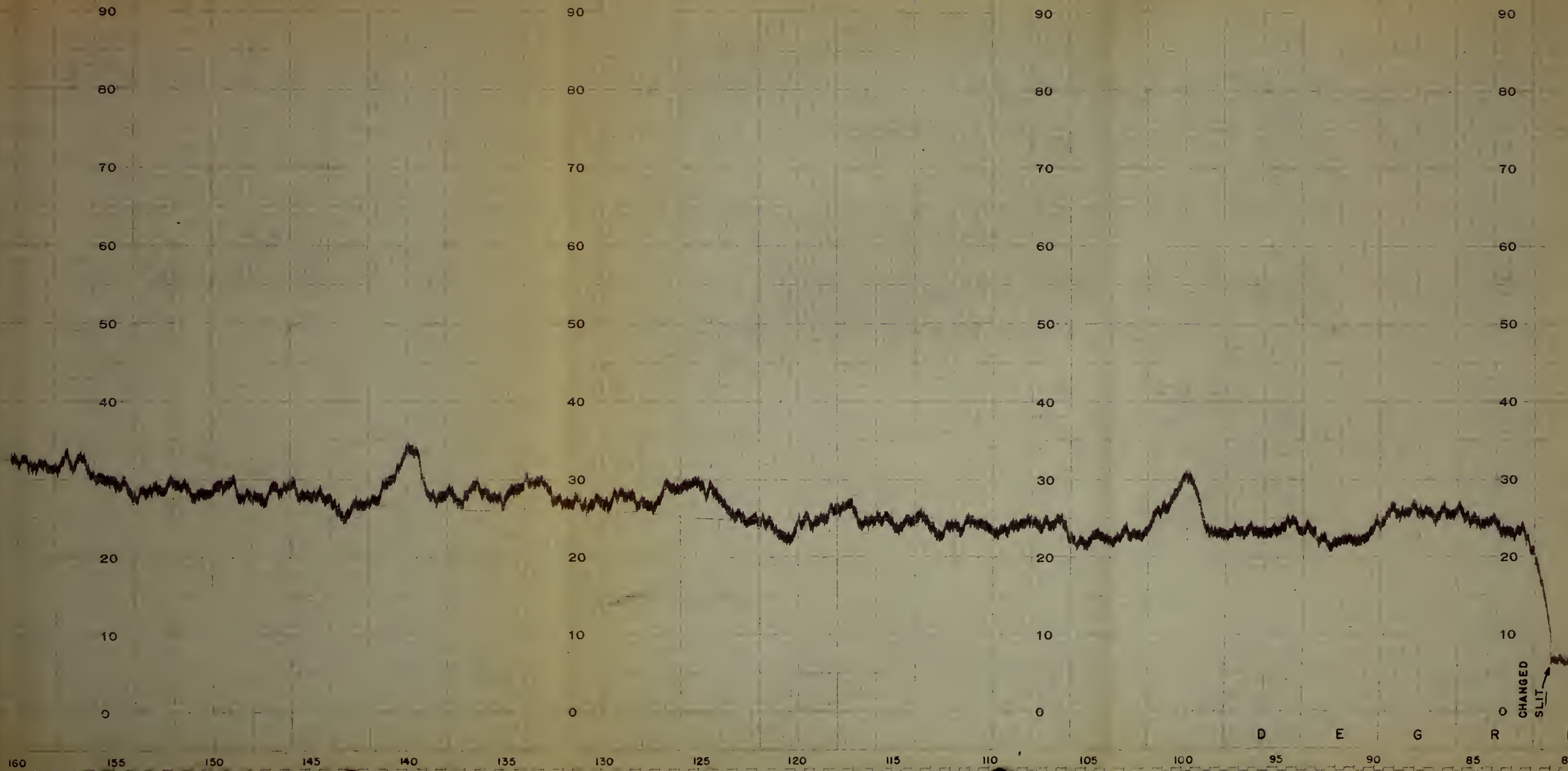
SPECTROMETER CHARTS

The following fifteen charts are reproductions of the actual traces just as they came from the machine except for titles, notes and degrees 2θ which were added afterward.

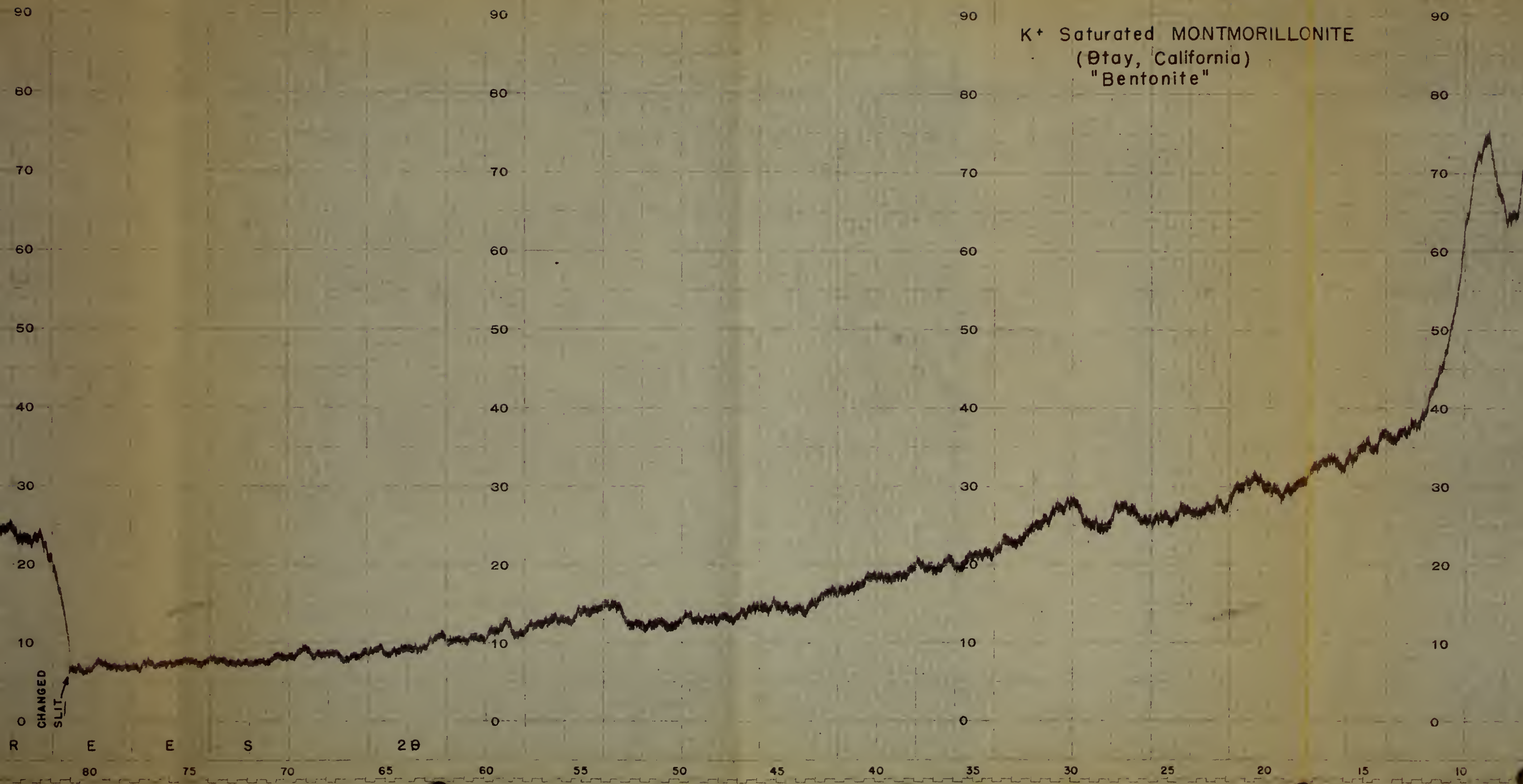


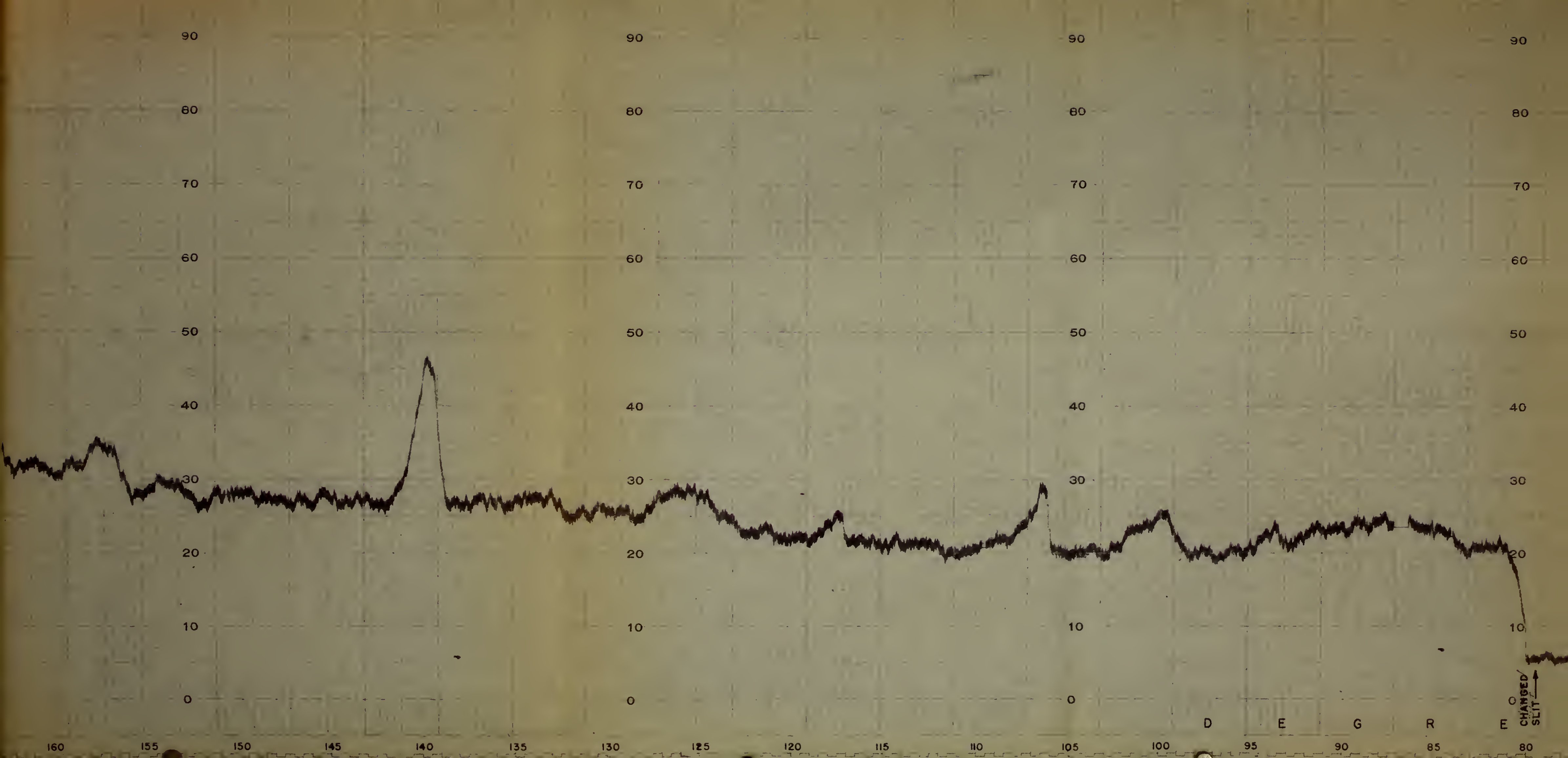
H⁺ Saturated MONTMORILLONITE
(Otay, California)
"Bentonite"



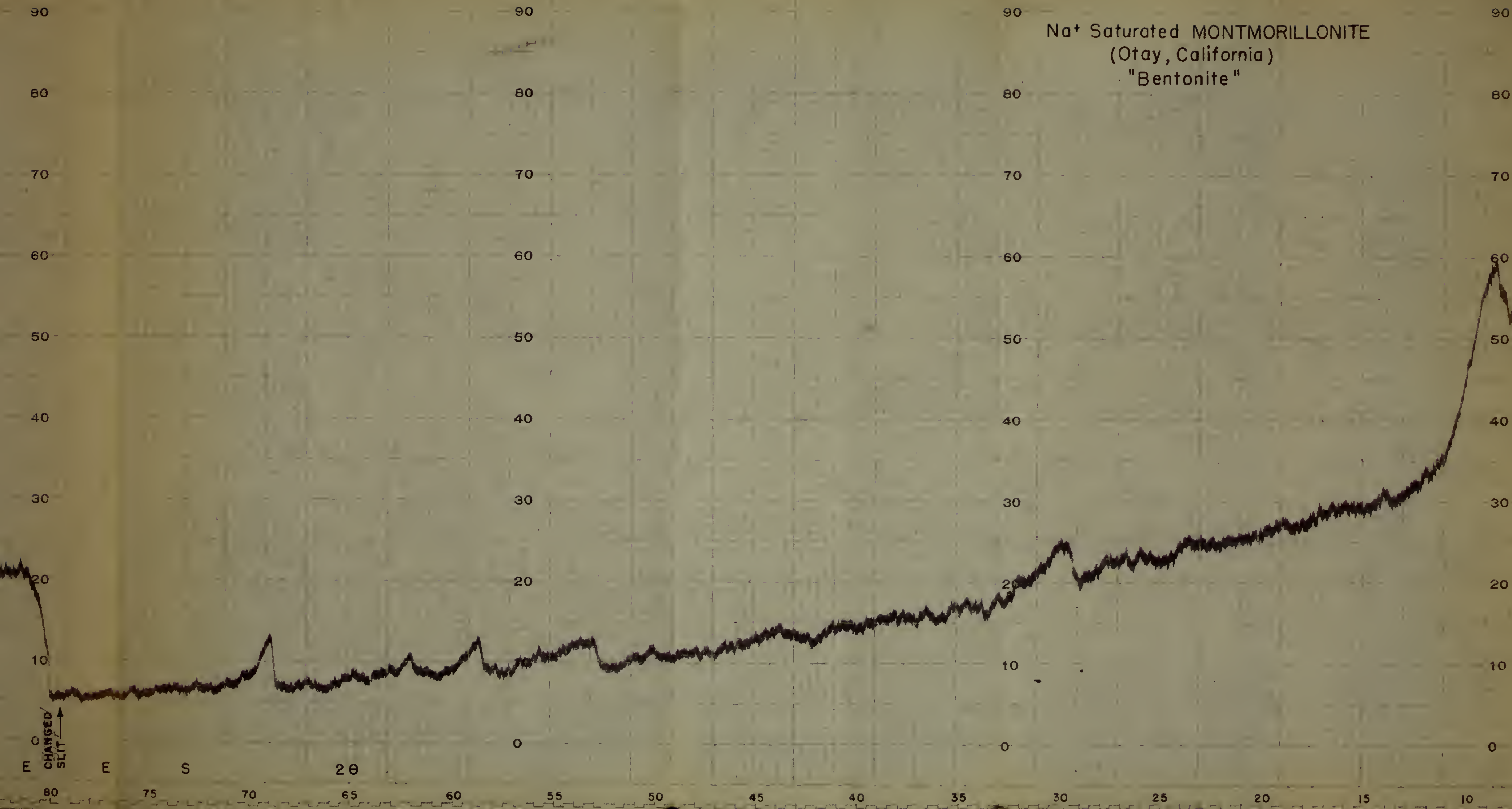


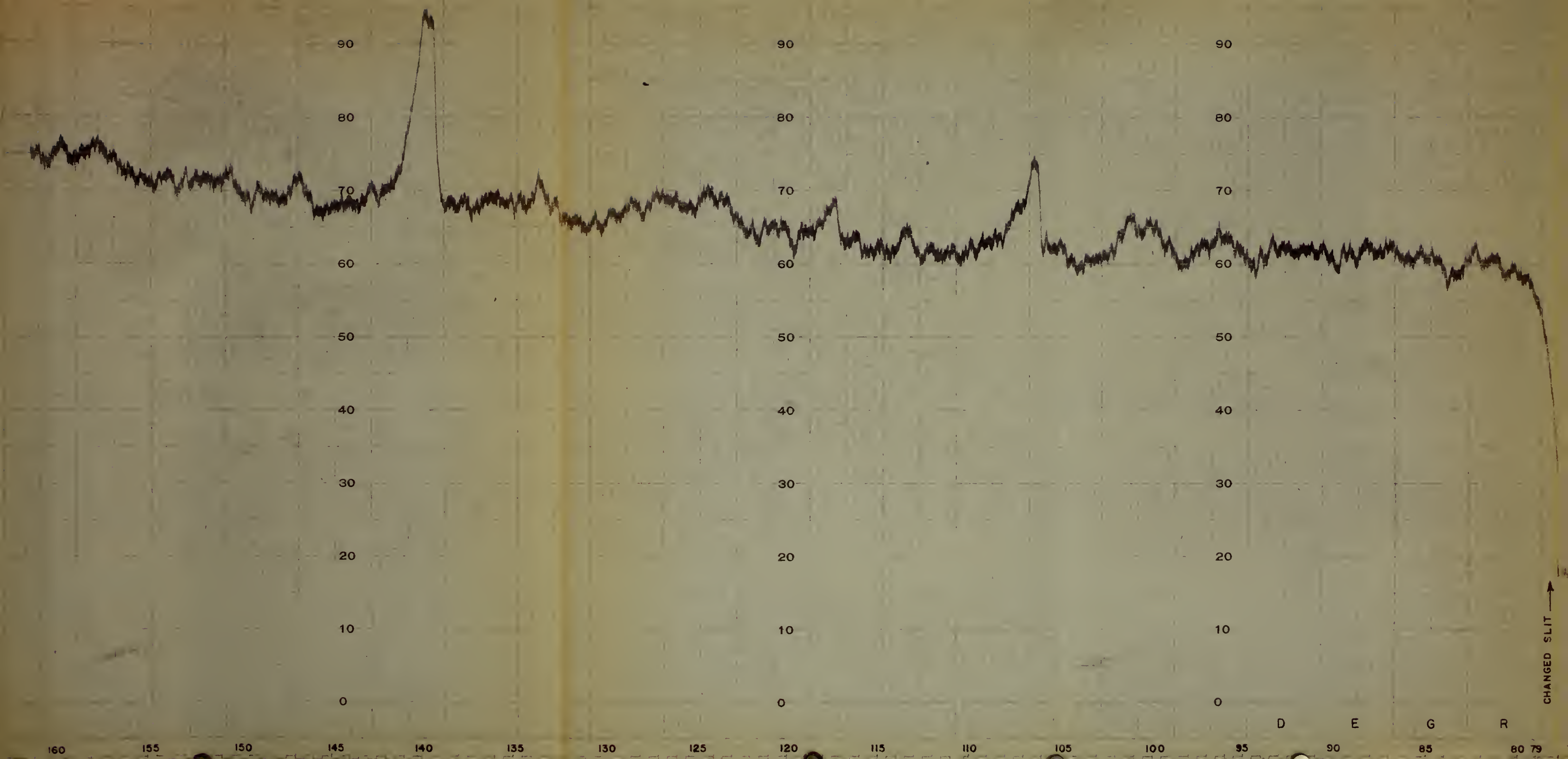
K⁺ Saturated MONTMORILLONITE
(Otay, California)
"Bentonite"



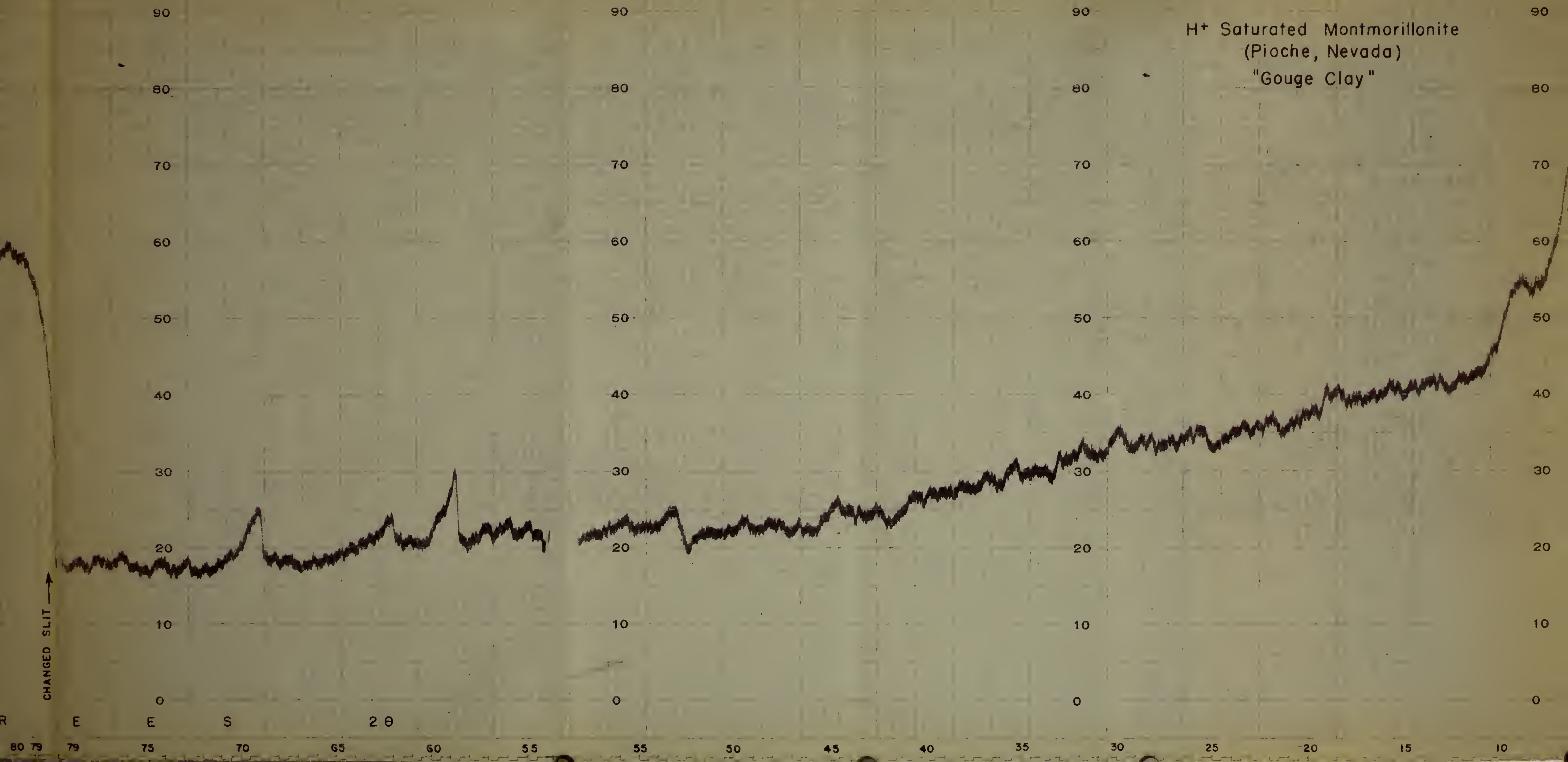


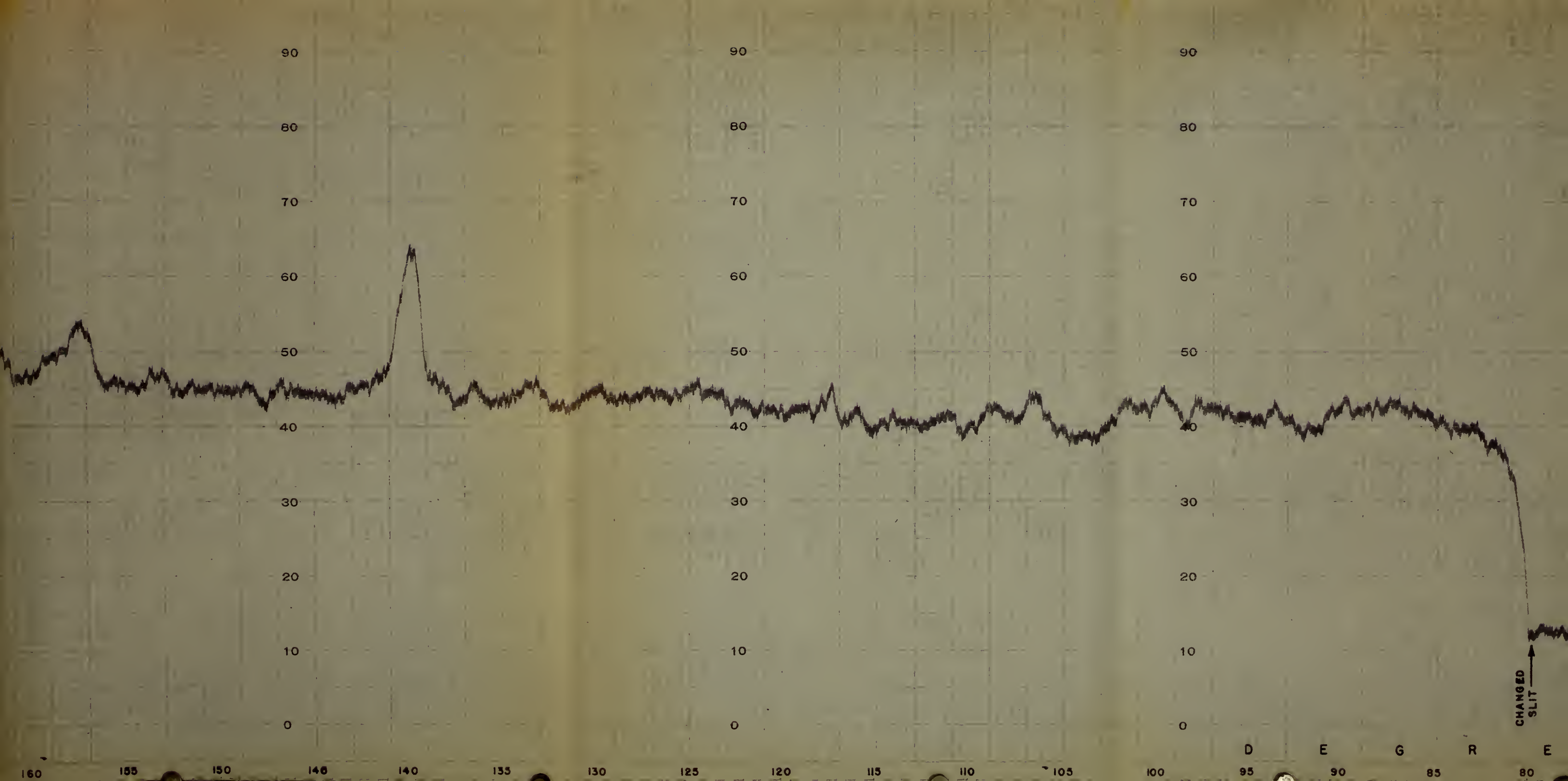
Na⁺ Saturated MONTMORILLONITE
(Otay, California)
"Bentonite"



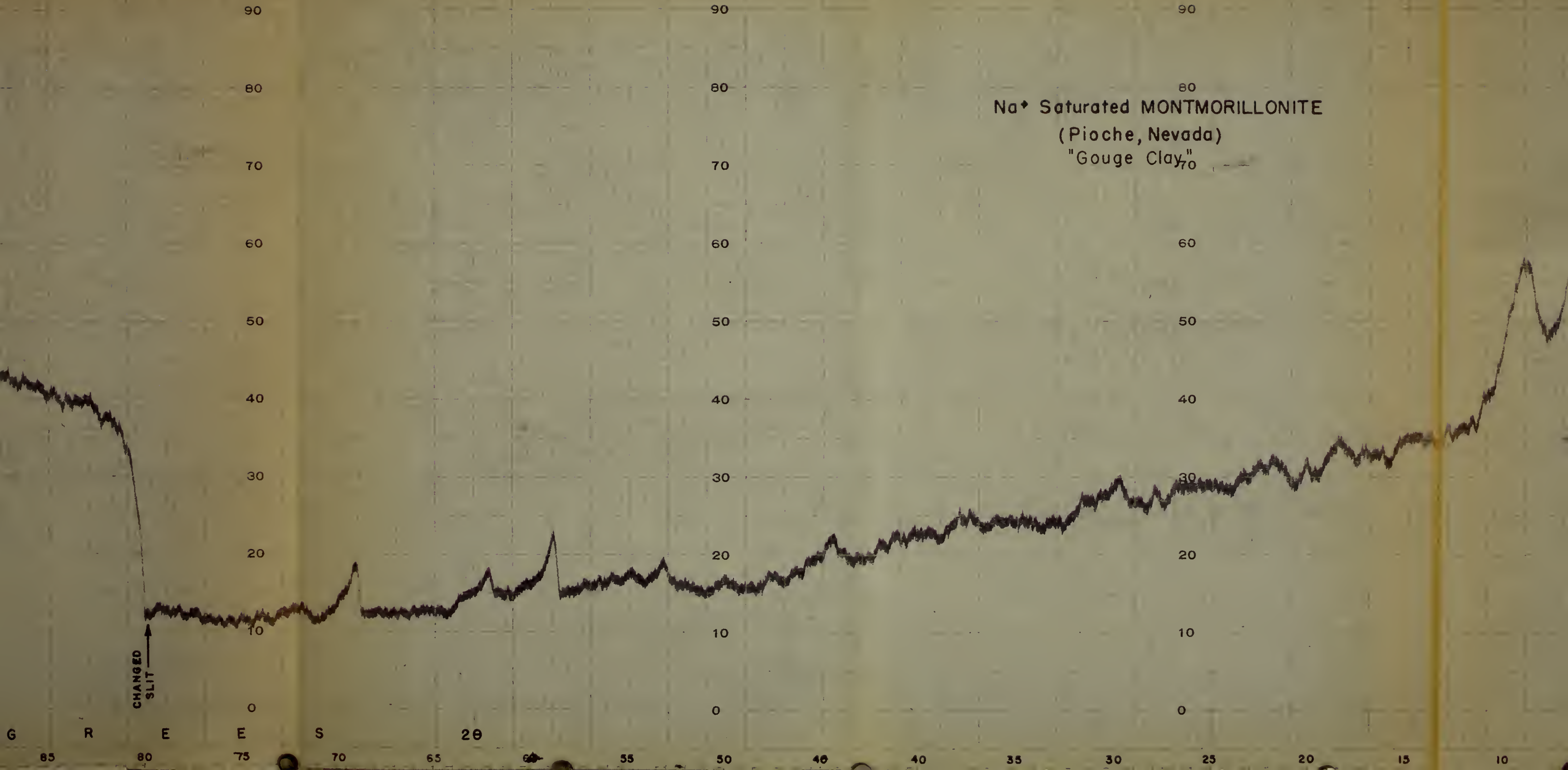


H⁺ Saturated Montmorillonite
(Pioche, Nevada)
"Gouge Clay"





Na⁺ Saturated MONTMORILLONITE
(Pioche, Nevada)
"Gouge Clay"



RENEW CHART

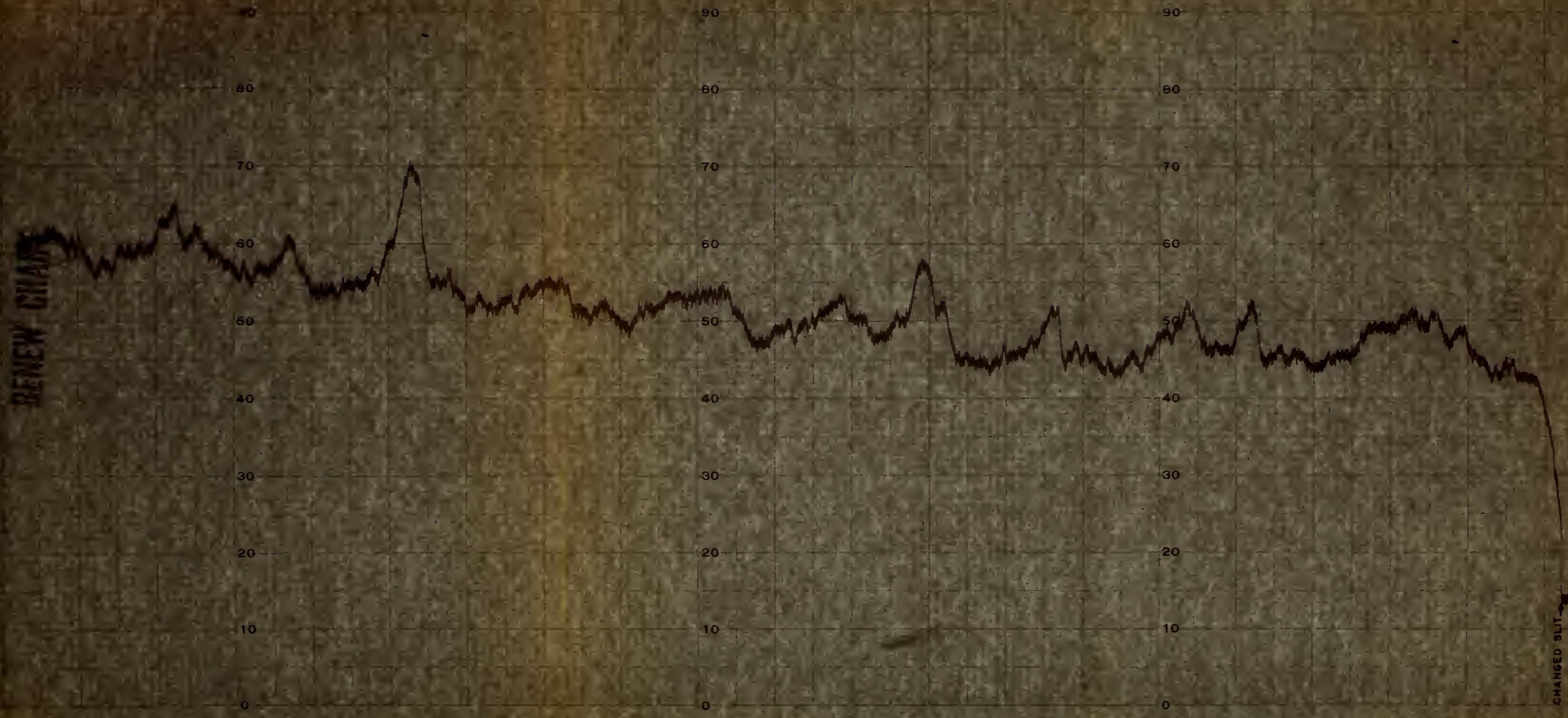
90
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90
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50
40
30
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0

90
80
70
60
50
40
30
20
10
0

140 135 130 125 120 115 110 105 100 95 90 85 80

CHANGED SLIT



H⁺ Saturated ILLITE
(Joliet, Illinois)

RENEW CHART

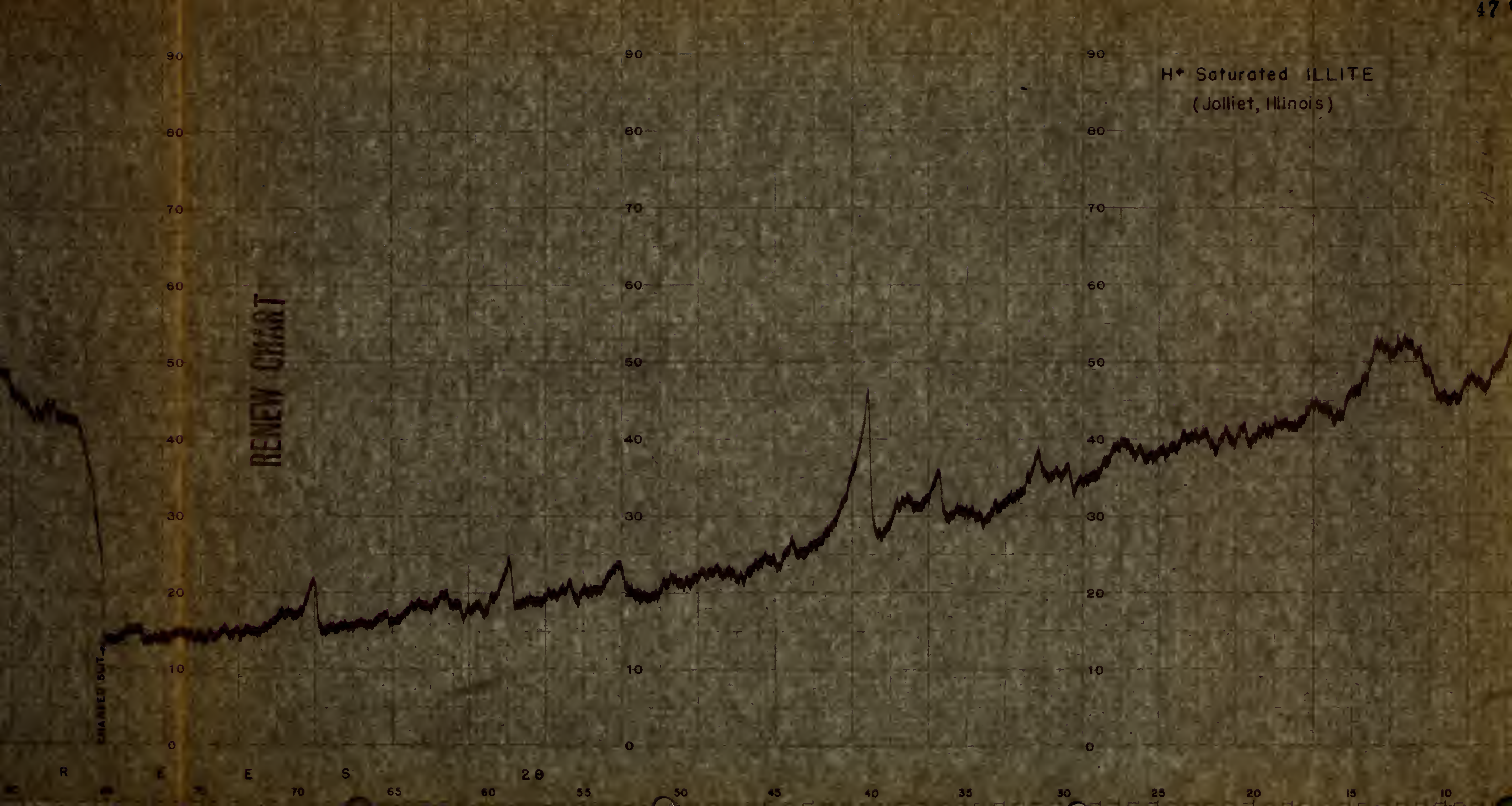
CHARTED SUT-4

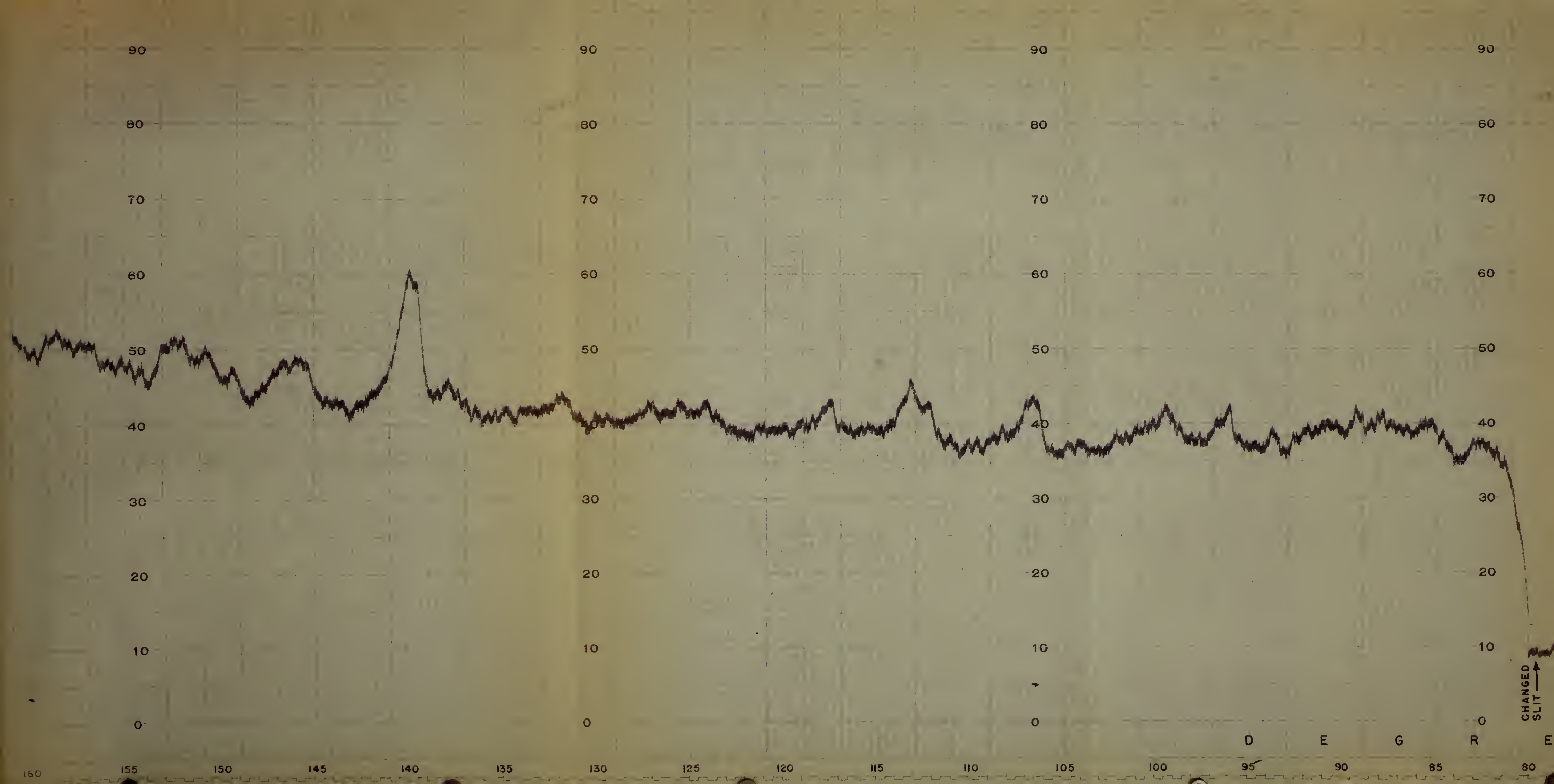
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90
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20
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0

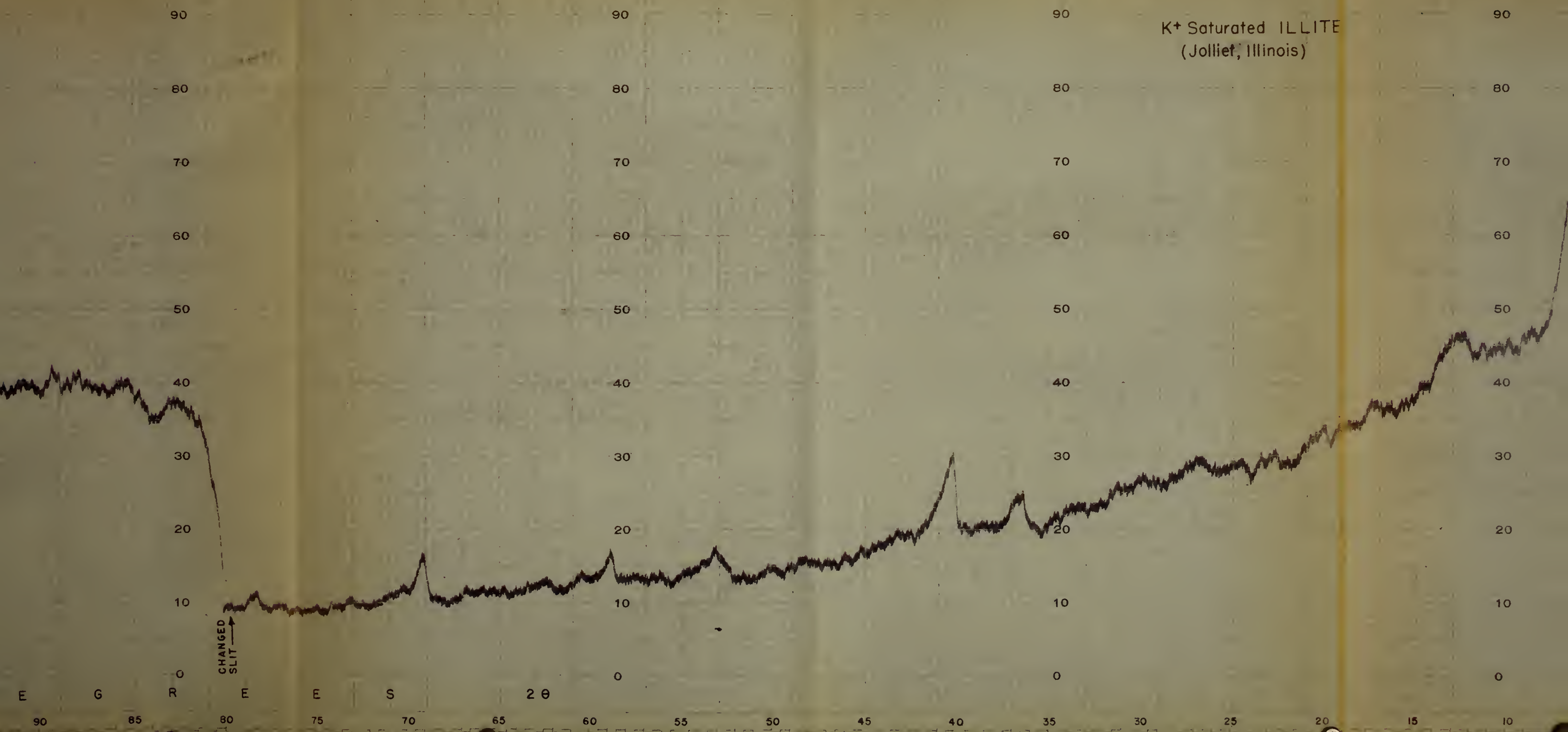
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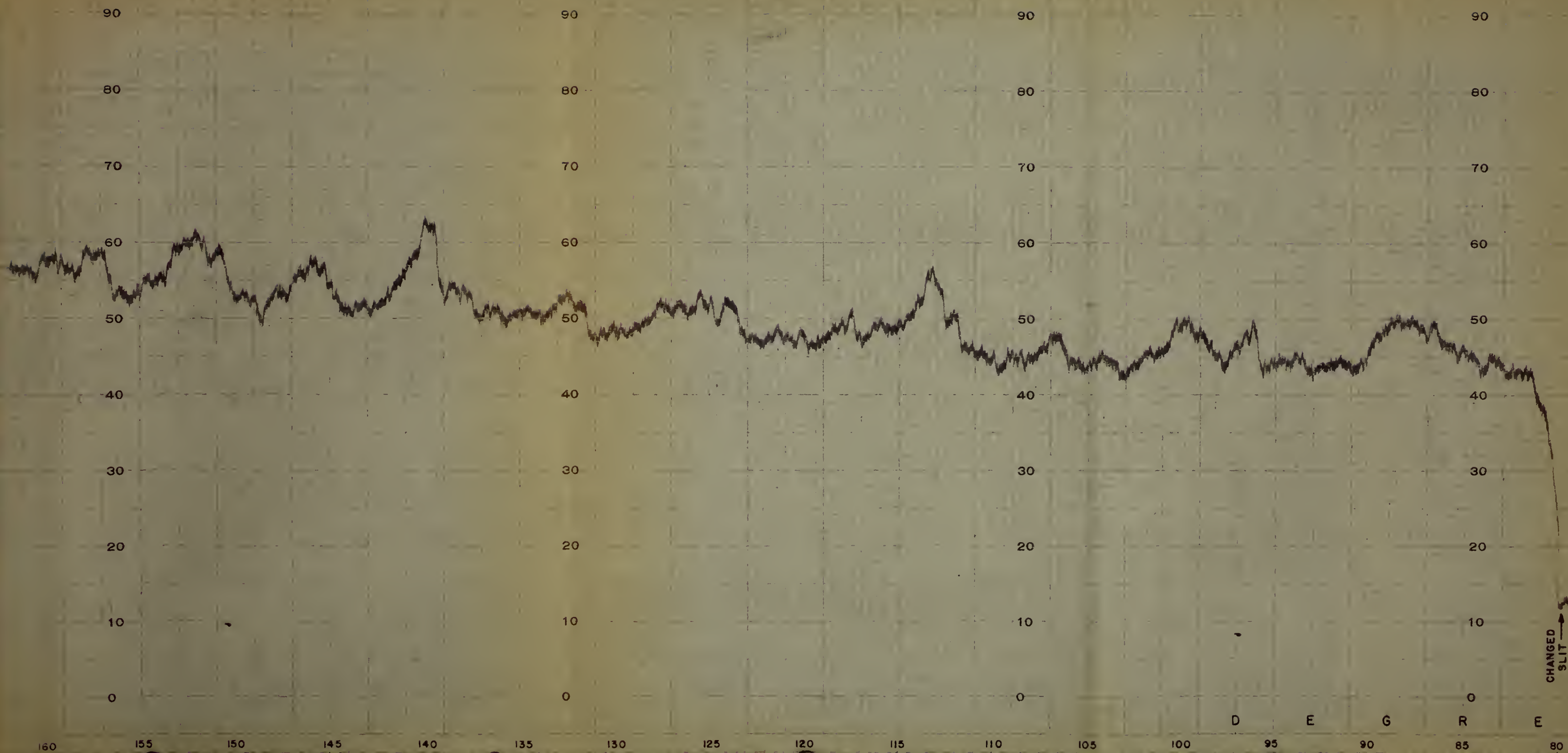
R E E S 2θ 70 65 60 55 50 45 40 35 30 25 20 15 10

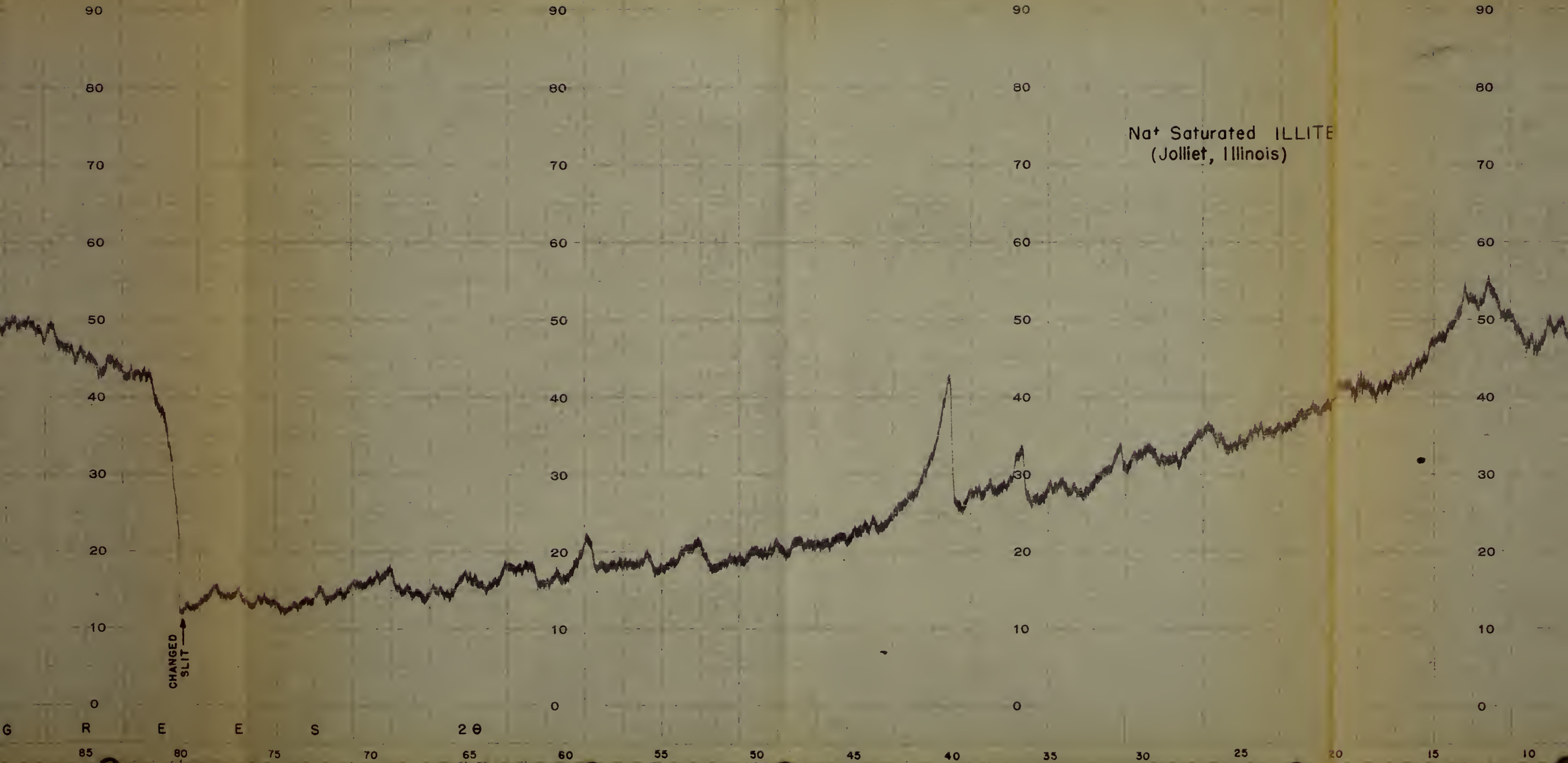




K⁺ Saturated ILLITE
(Joliet, Illinois)







90
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10
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90
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70
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90
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30
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10
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90
80
70
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30
20
10
0

160

155

150

145

140

135

130

125

120

115

110

105

100

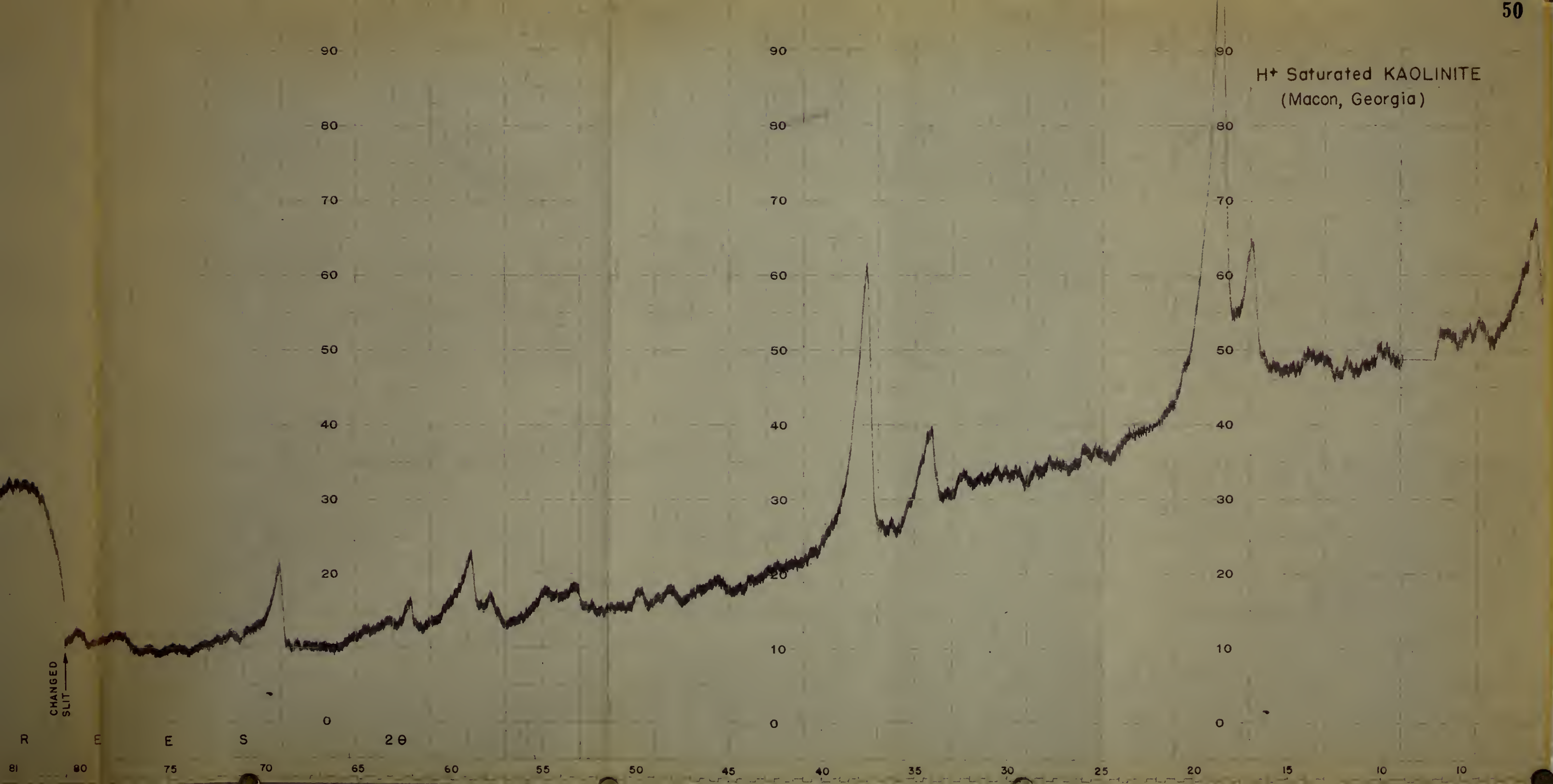
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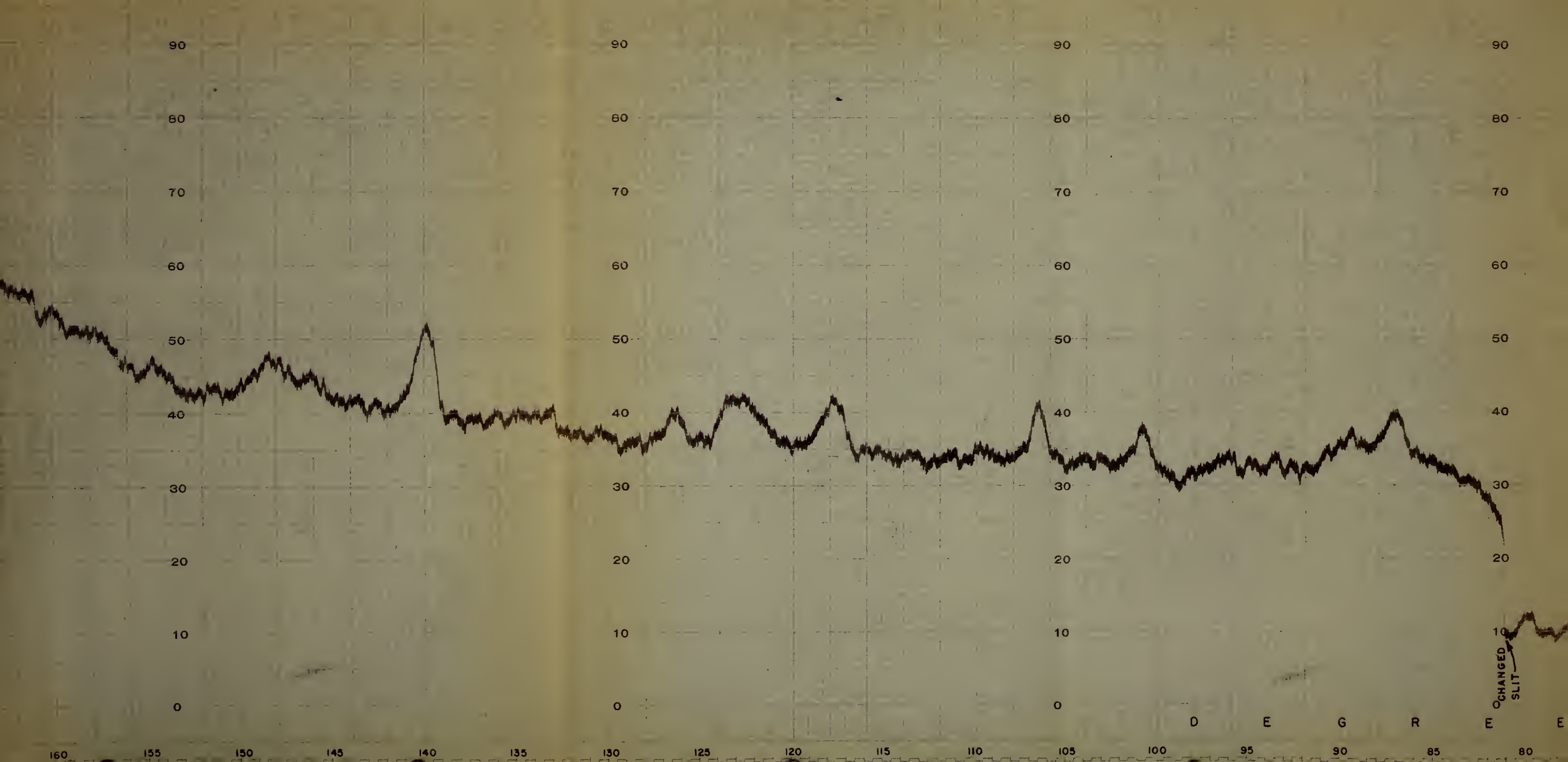
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85

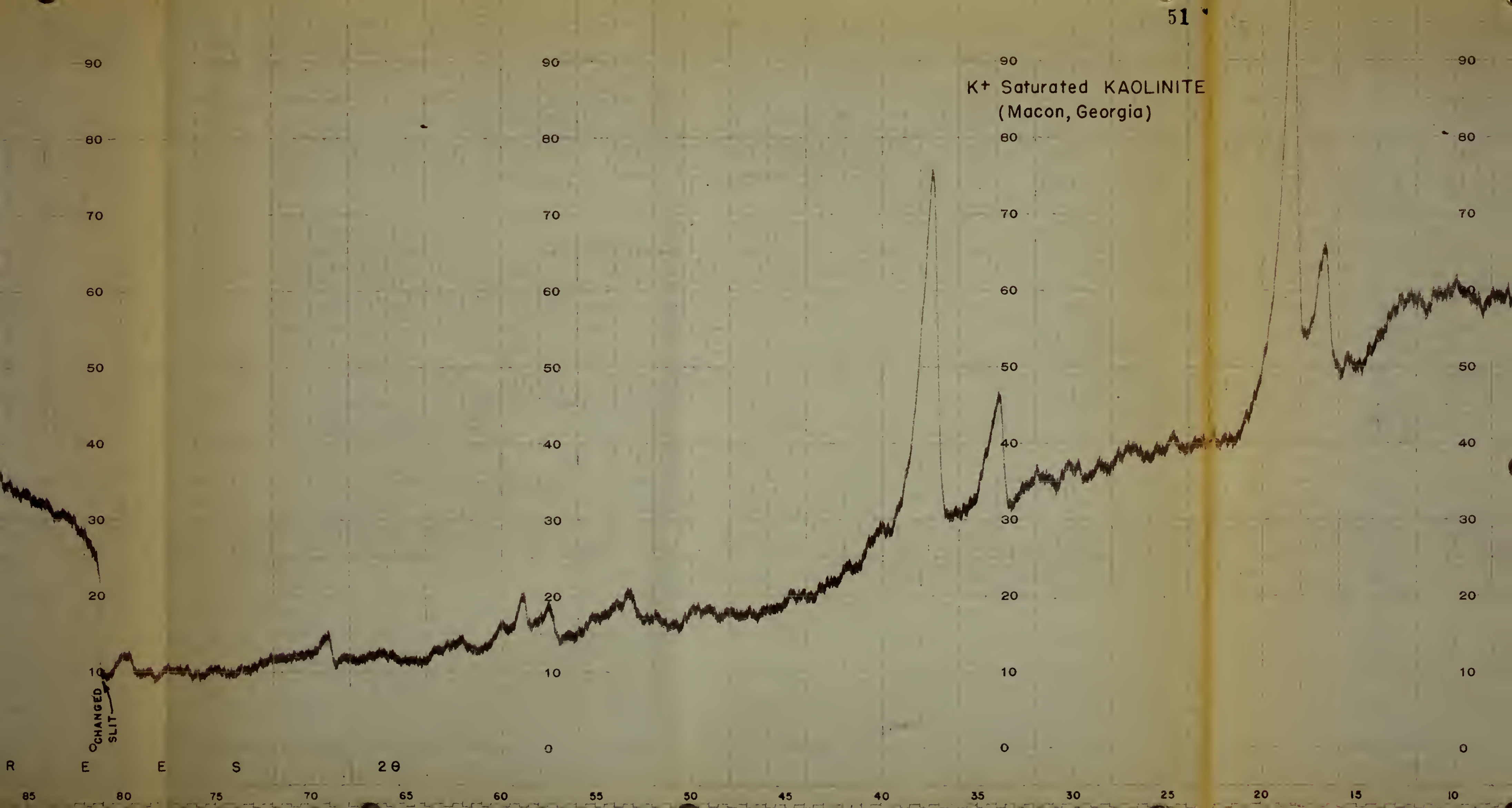
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CHANGED
SLIT





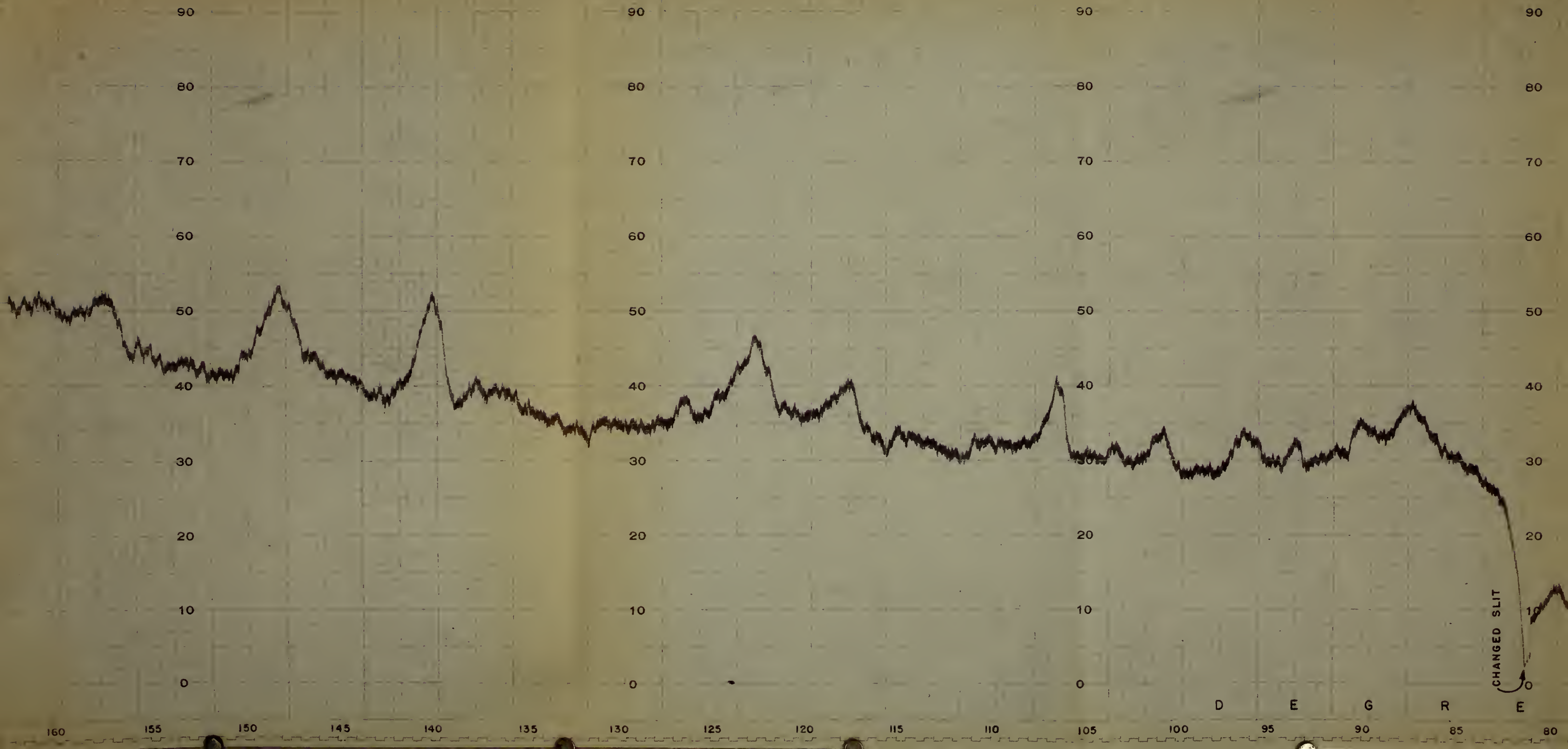
K⁺ Saturated KAOLINITE
(Macon, Georgia)



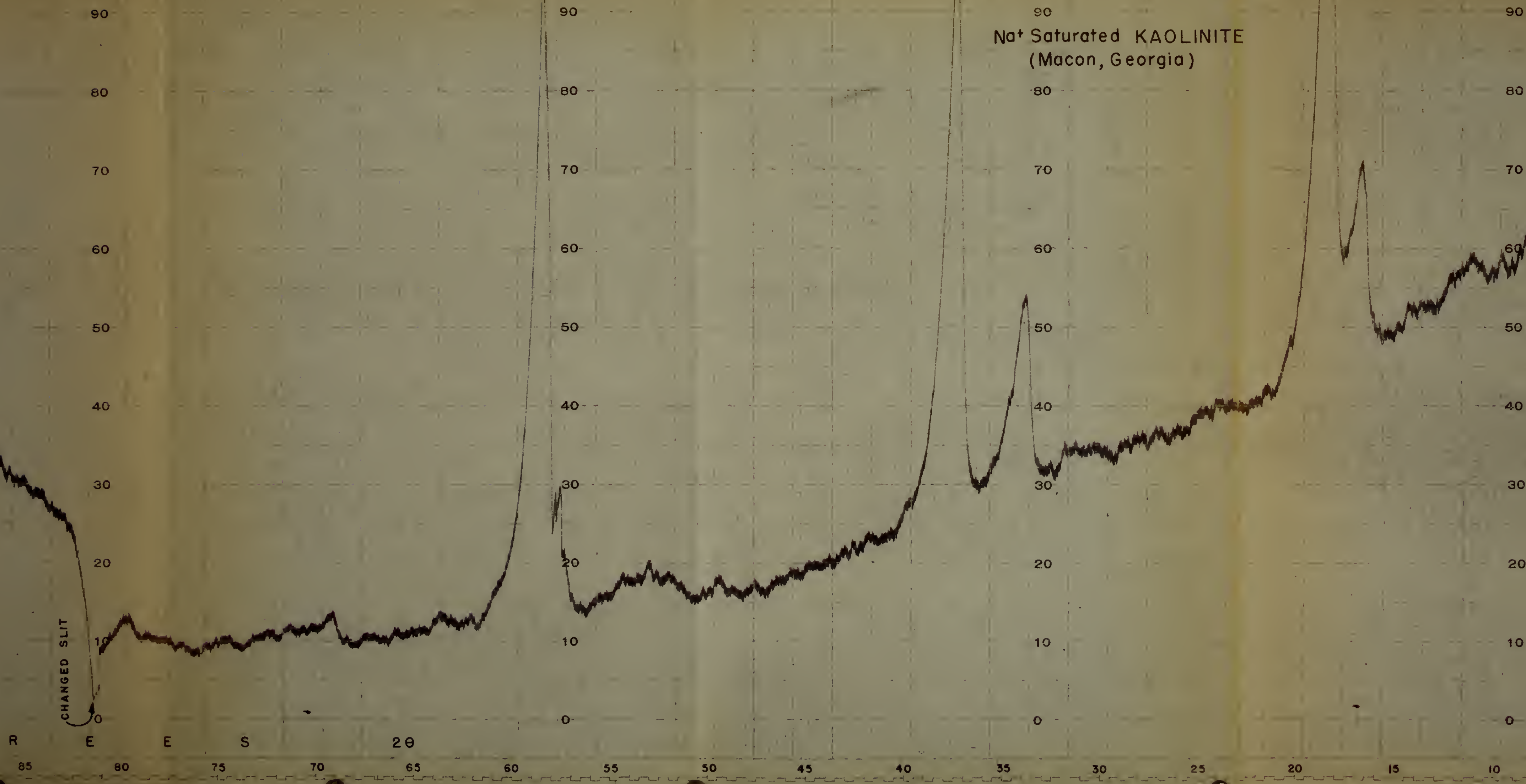
CHANGED
SLIT

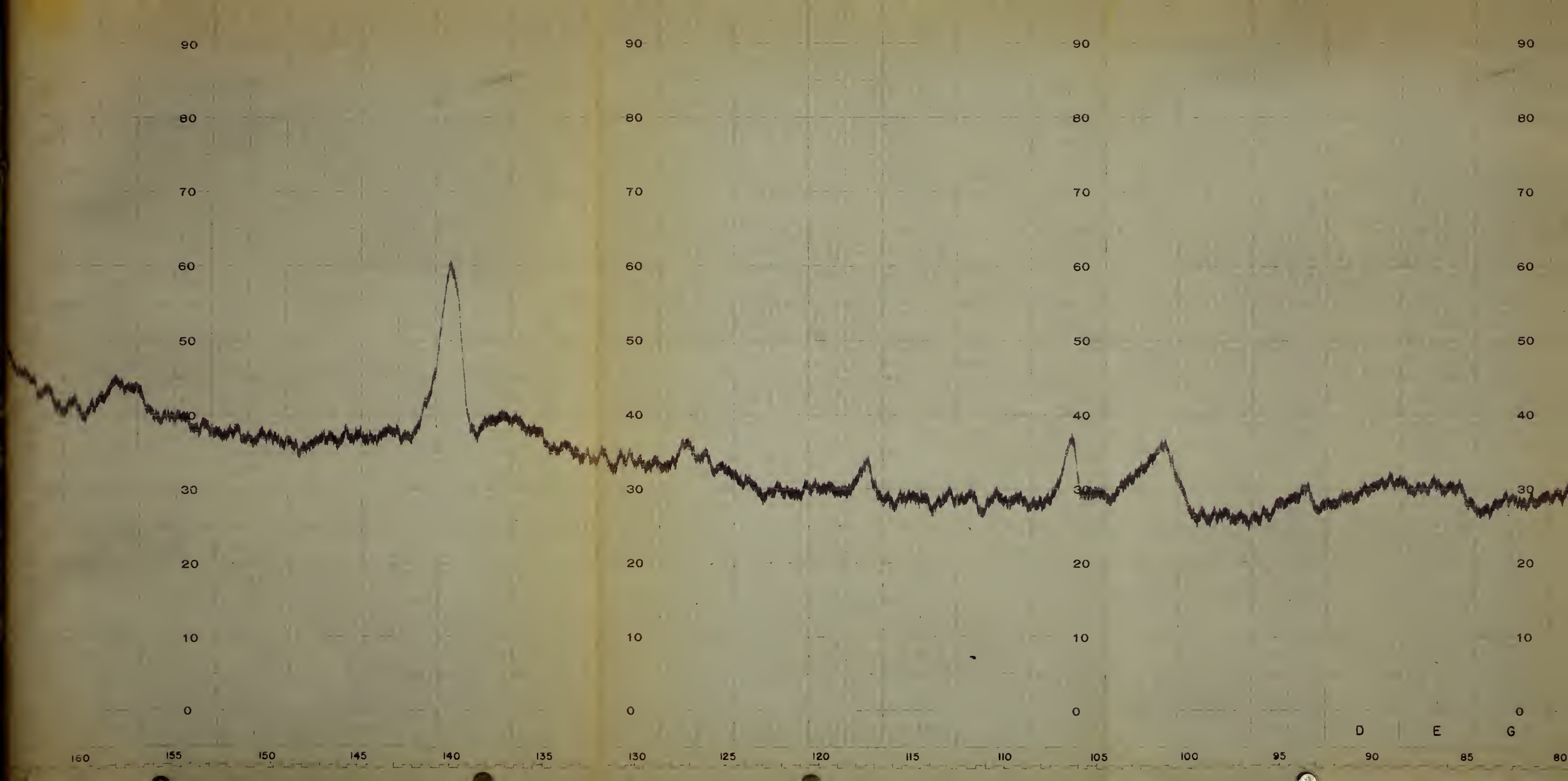
R E S 2θ

85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10

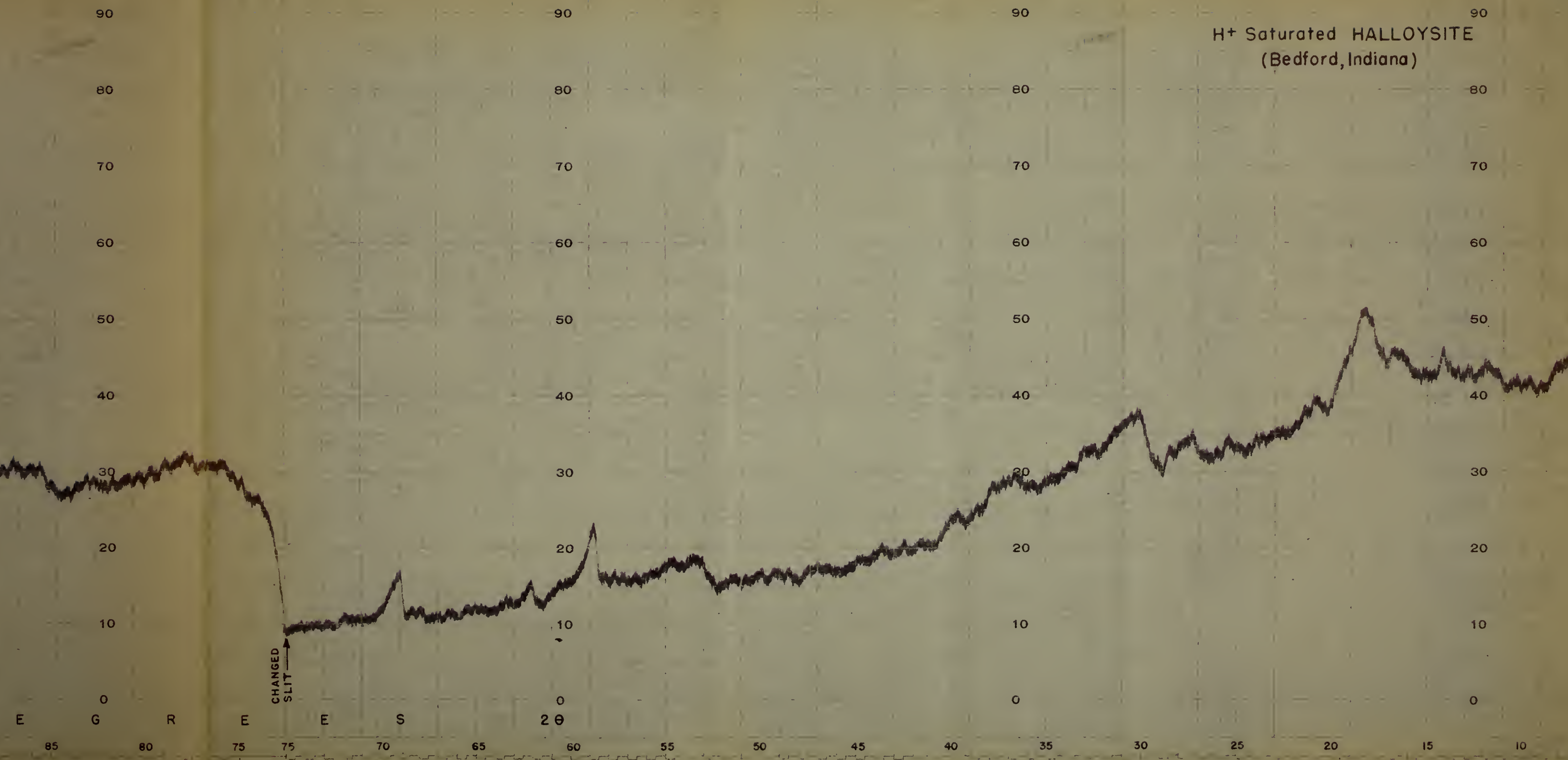


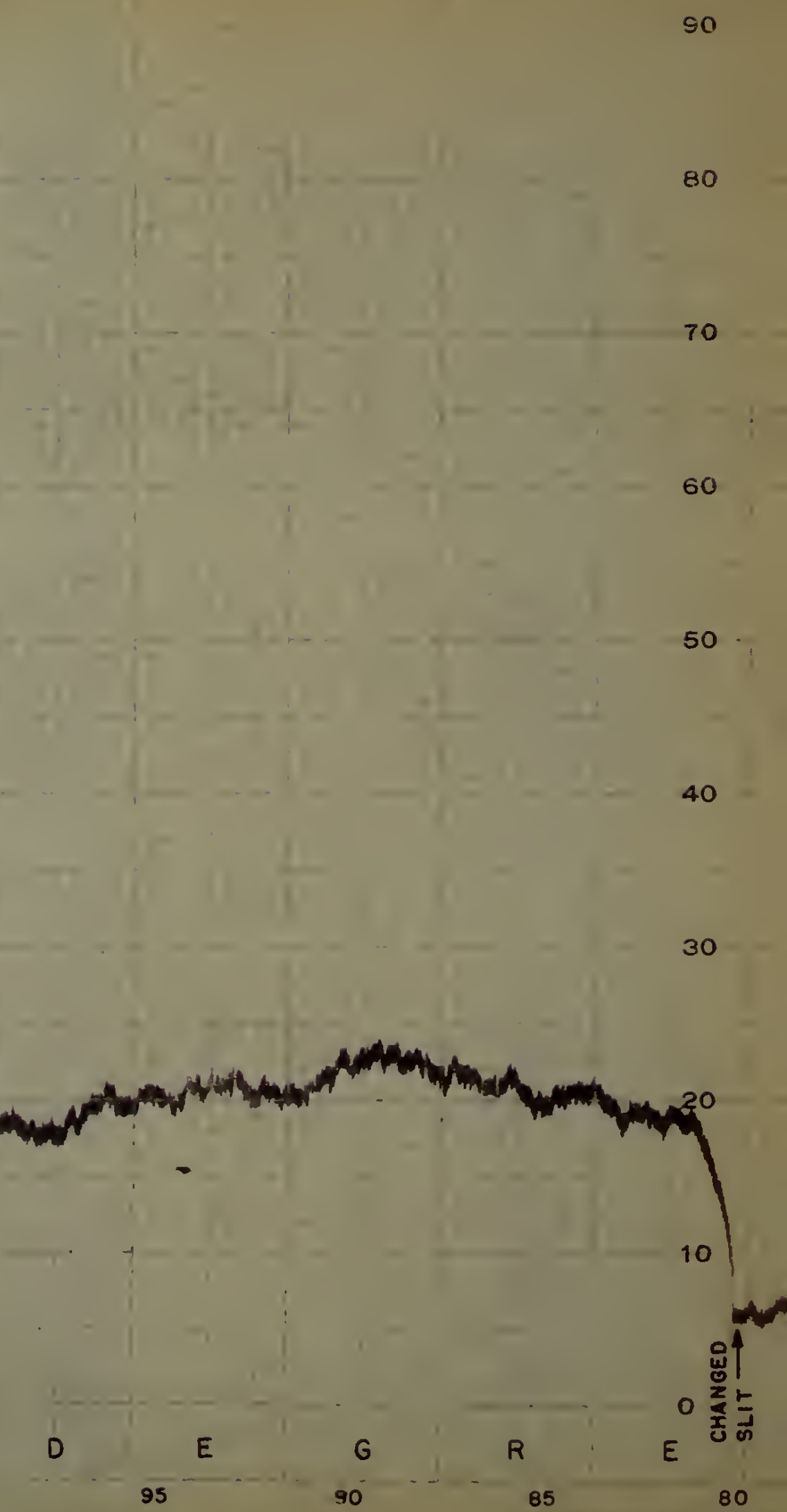
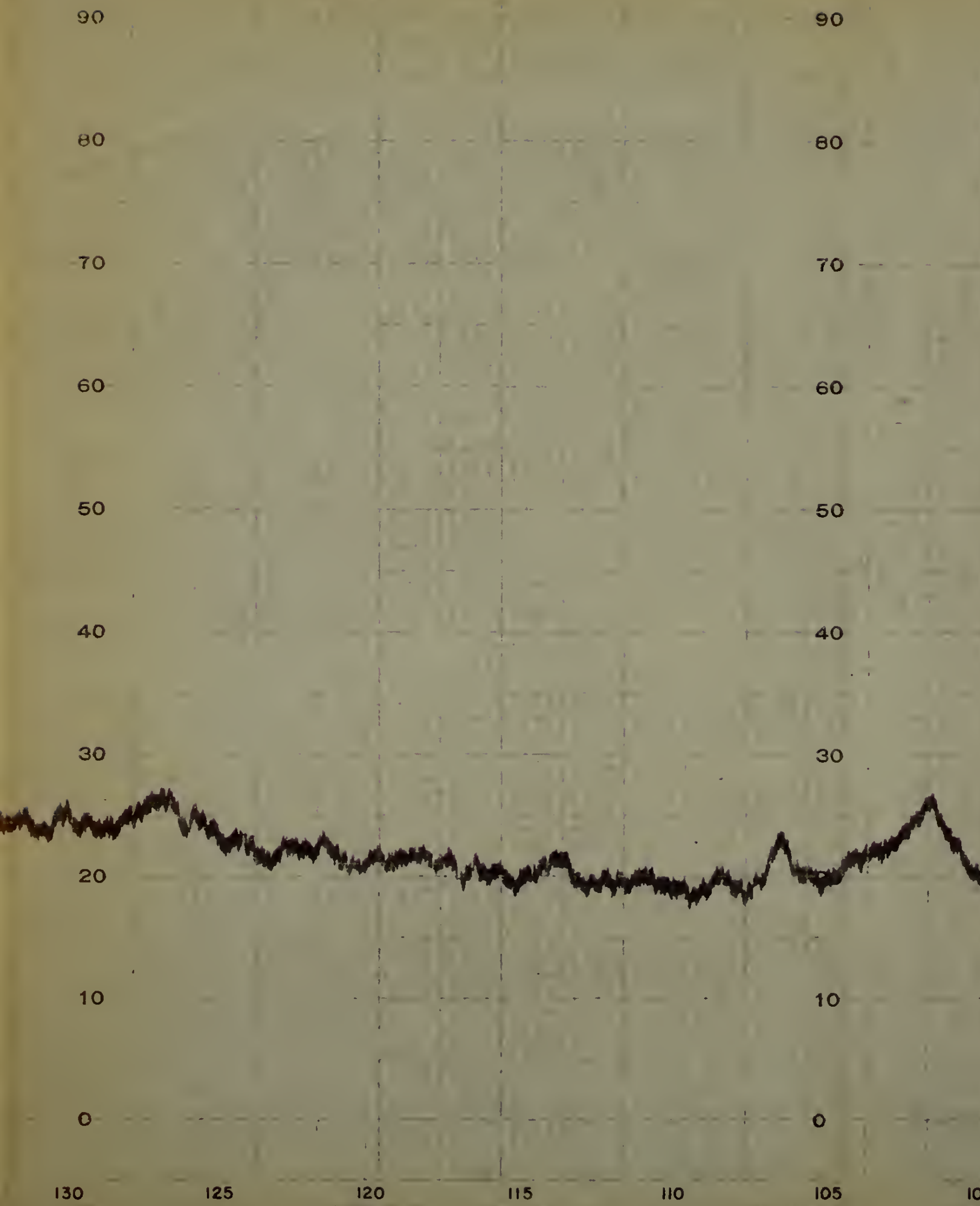
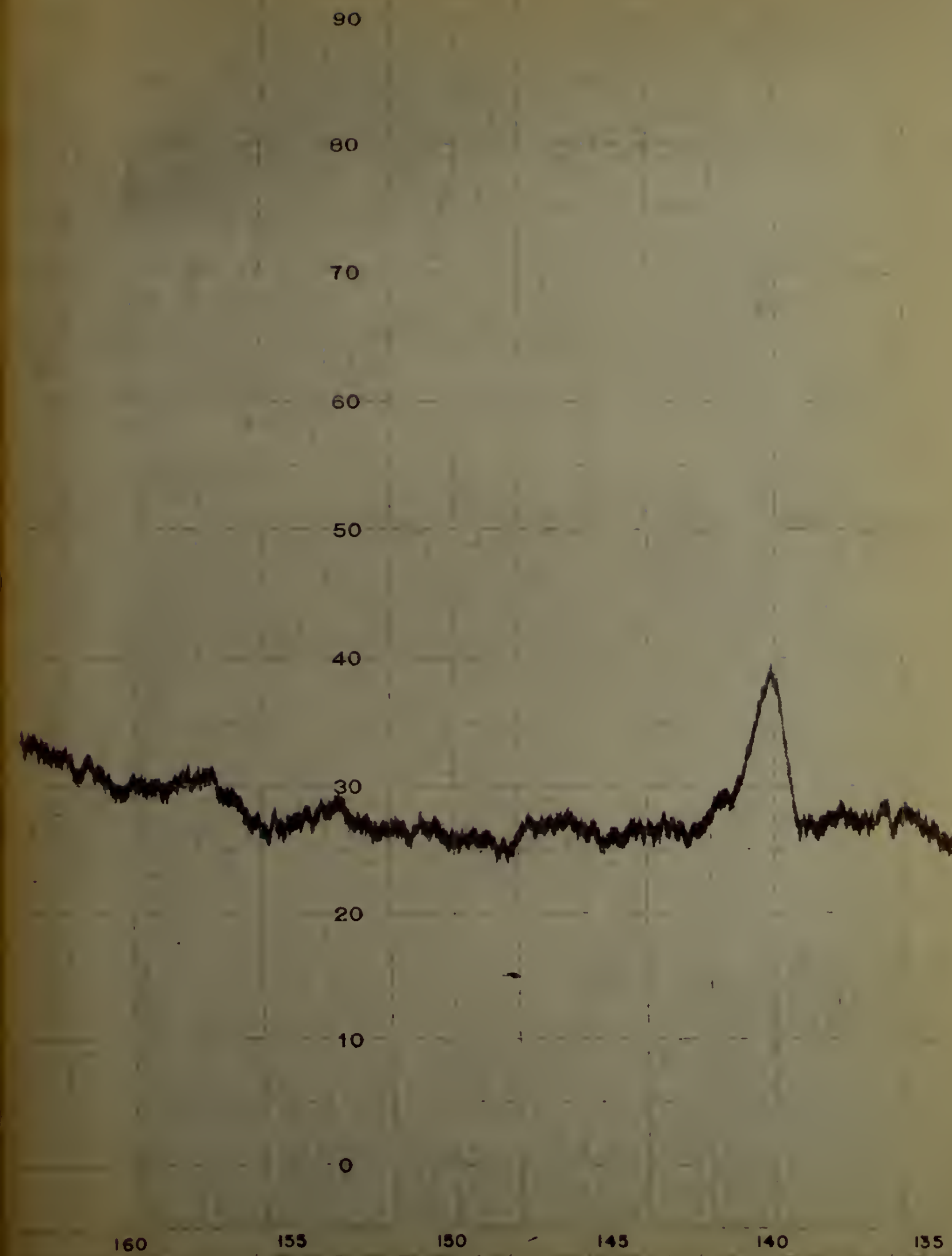
Na⁺ Saturated KAOLINITE
(Macon, Georgia)



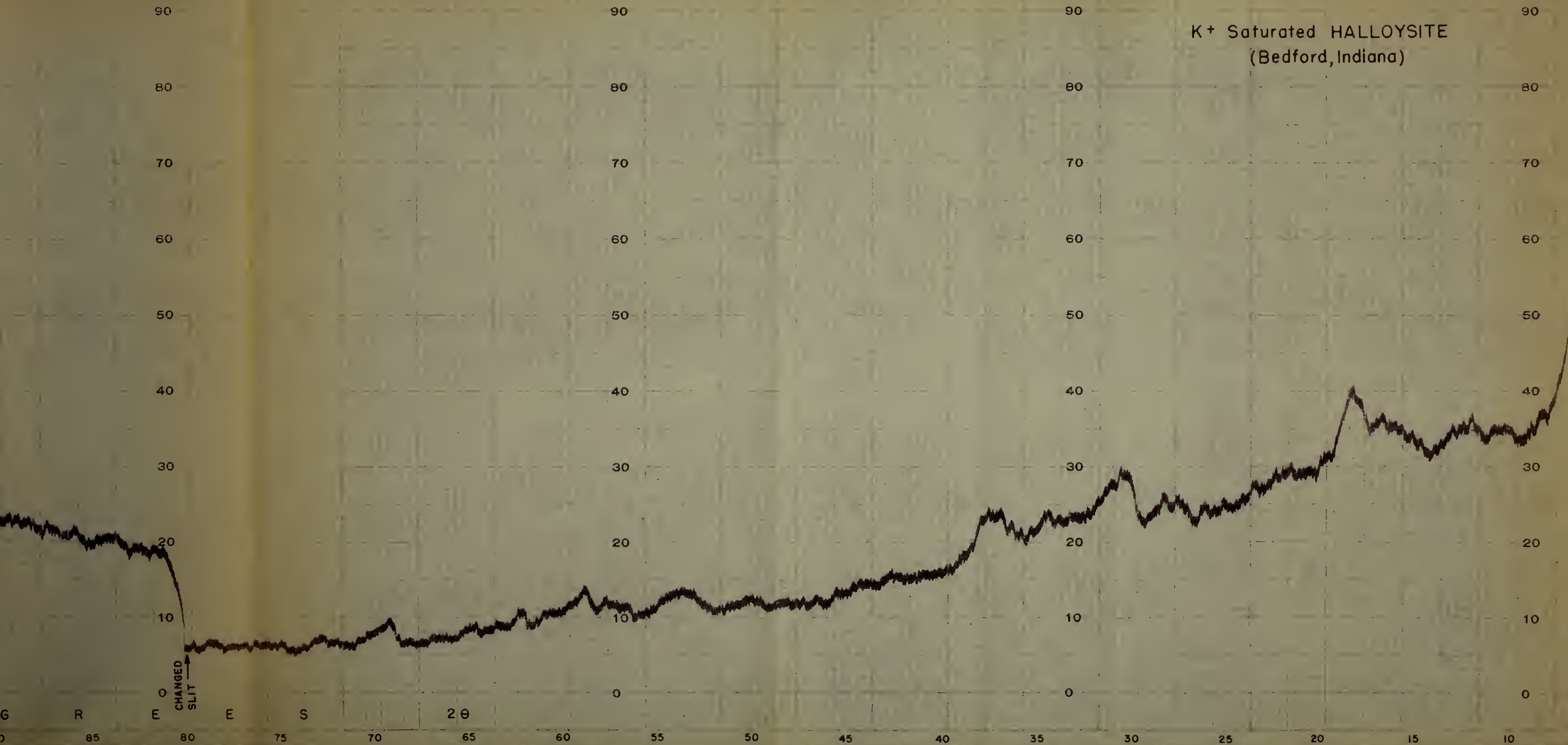


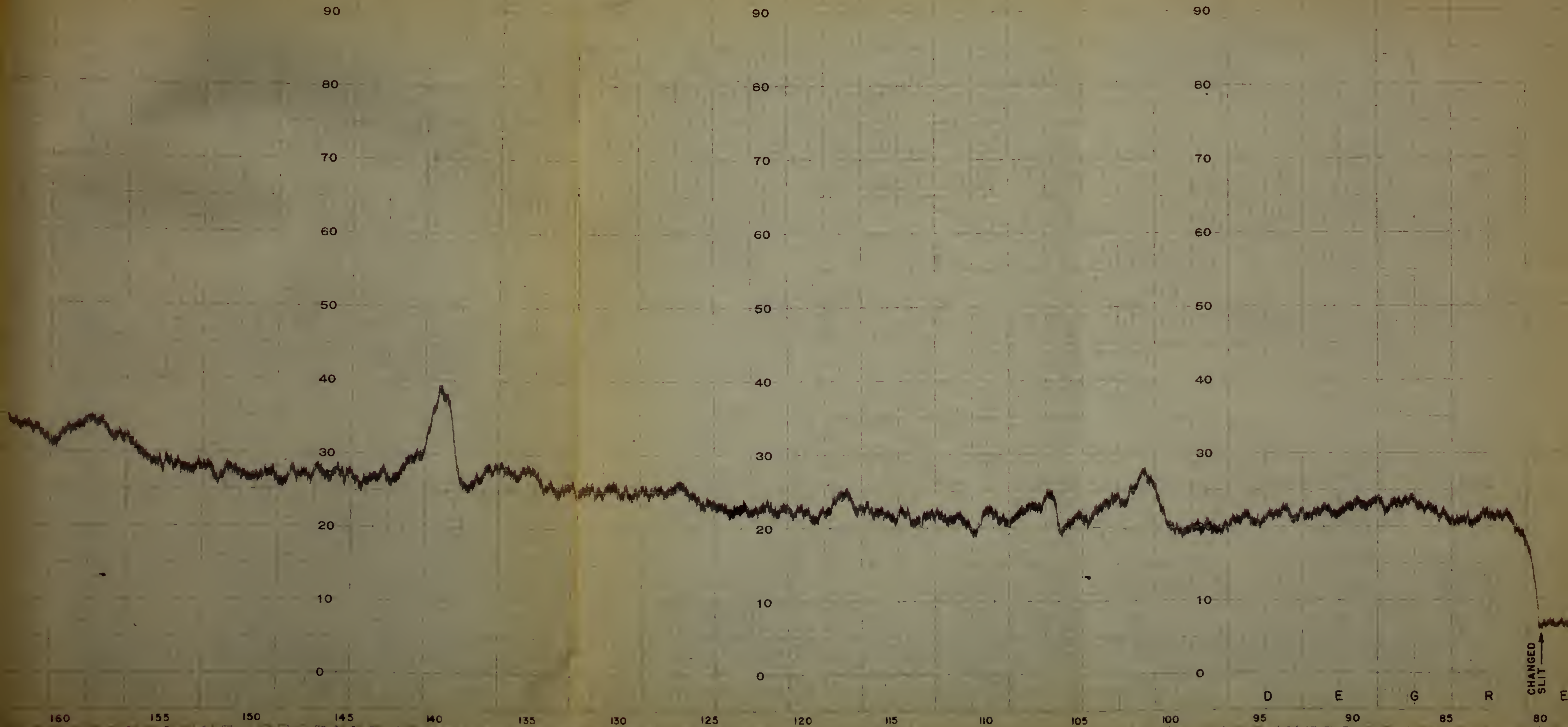
H⁺ Saturated HALLOYSITE
(Bedford, Indiana)



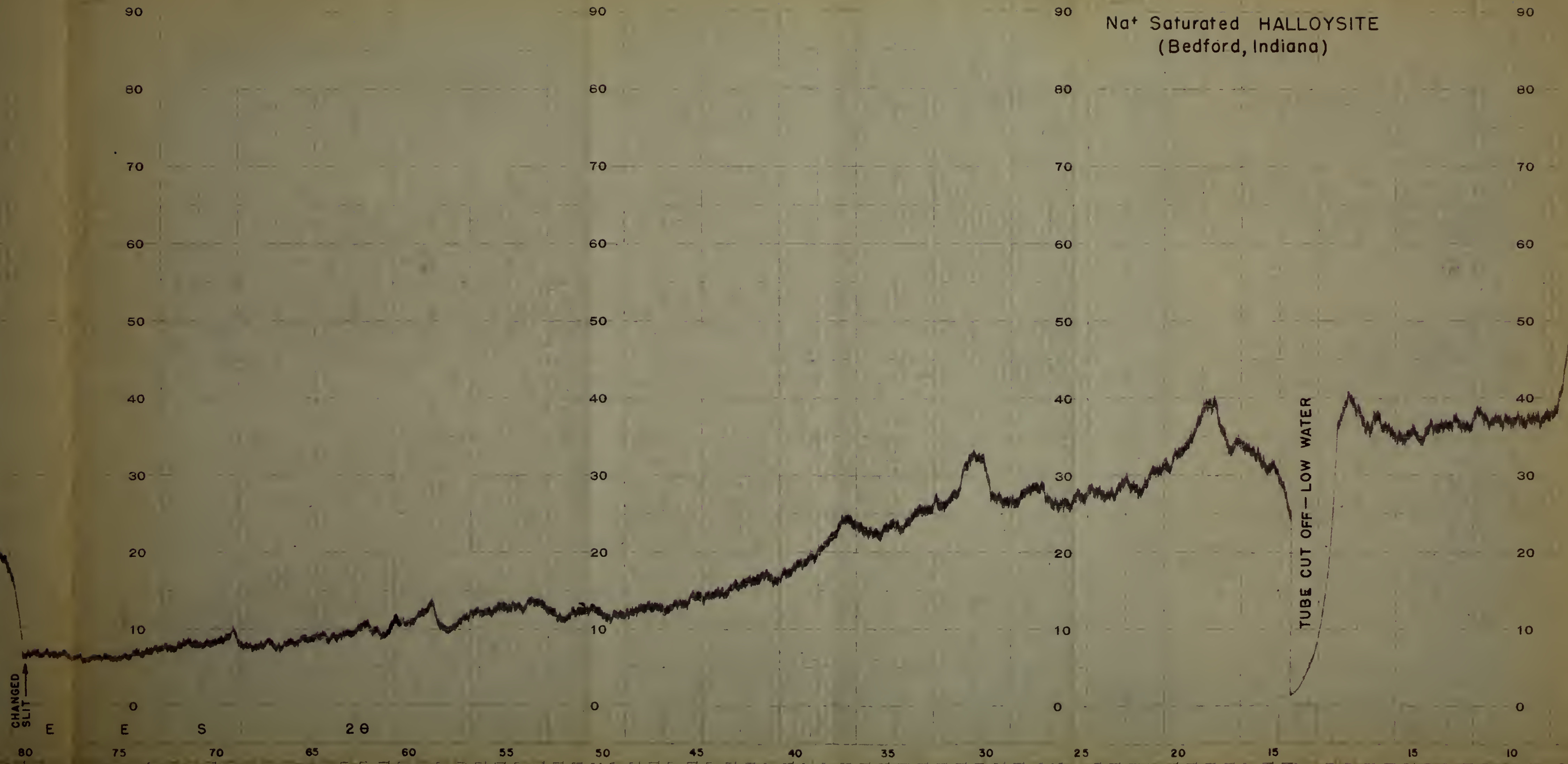


K⁺ Saturated HALLOYSITE
(Bedford, Indiana)





Na⁺ Saturated HALLOYSITE
(Bedford, Indiana)



APPENDIX C

FILM DIFFRACTION PATTERNS

<u>Otay, California - Montmorillonite (Bentonite) - H^+ Saturated</u>										
<u>Ring No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>
$d(\text{\AA})$	4.67	3.69	3.47	2.90	2.61	2.29	2.02	1.69	1.56	1.27

<u>Pioche, Nevada - Montmorillonite (Gouge Clay) - Na^+ Saturated</u>												
<u>Ring No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
$d(\text{\AA})$	4.42	3.59	3.21	2.56	2.49	2.12	1.79	1.65	1.51	1.49	1.26	1.24

<u>Joliet, Illinois - Illite - H^+ Saturated</u>													
<u>Ring No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>
$d(\text{\AA})$	4.94	4.53	4.20	4.13	3.33	2.57	2.12	1.81	1.53	1.49	1.44	1.37	1.19

<u>Macon, Georgia - Kaolinite - H^+ Saturated</u>												
<u>Ring No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
$d(\text{\AA})$	4.42	3.65	2.58	2.35	2.02	1.95	1.91	1.85	1.80	1.68	1.59	1.55

<u>Bedford, Indiana - Halloysite - K^+ Saturated</u>											
<u>Ring No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>
$d(\text{\AA})$	4.75	4.38	3.59	2.56	2.34	2.22	1.79	1.46	1.37	1.29	1.22

APPENDIX D

COMPARISON OF COMPUTED INTERPLANAR
SPACINGS WITH A.S.T.M. INDEX CARDS

Otay, California - Montmorillonite (Bentonite)

Diffraction Pattern

Intensity	Order of Intensity	Interplanar Spacing (Å)
188	1	15.25
2	12	9.24
5	9	5.05
4	10	4.84
28	4	4.42
22	6	2.54
13		2.34#
4	11	2.21
18		2.02#
10	7	1.57
30	3	1.50
27	5	1.43
8	8	1.34
36	2	1.29
84		1.22#

These lines are the Hannawalt Index for pure Aluminum Metal from the sample holder and are therefore excluded from the order of intensity count.

ASTM Card for Montmorillonite (API - 19)

d	15.26	1.50	5.16	15.26	d in Å	I/I ₁	d in Å	I/I ₁
					λ = 1.539			
I/I ₁	100	80	50	100	15.26	100		
					5.16	50		
					4.50	30		
					3.87	10		
					3.33	10		
					3.07	10		
					2.24	10		
					1.69	30		
					1.50	80		
					1.29	30		
					1.24	30		
					1.12	10		
					1.03	10		
					0.98	10		
					0.87	10		
					Diffuse Pattern			

(Al_{1.44}Fe_{.08}Mg_{.53}) (Al_{.10}Si_{3.90}) O₁₀

(OH)₂ Na_{.02} Ca_{.18})

Montmorillonite

Rad: Cu Kα

Filter: Nickel

Pioche, Nevada -- Montmorillonite (Gouge Clay)

Diffraction Pattern

Intensity	Order of Intensity	Interplanar Spacing (Å)
59	1	14.89
8	11	6.94
6	14	4.47
20	5	3.03
8	12	2.56
25		2.34#
10	8	2.22
32		2.02#
30	4	1.54
35	3	1.49
42	2	1.43
7	13	1.37
12	6	1.34
12	7	1.30
9	10	1.25
122		1.22#
10	9	1.19

These lines are the Hannawalt Index for pure Aluminum Metal from the sample holder and are therefore excluded from the order of intensity count.

ASTM Card for Montmorillonite (API - 16)

d	14.82	4.43	1.49	14.82	d in Å	I/I ₁	d in Å	I/I ₁
					λ = 1.539			
I/I ₁	100	80	80	100	14.82	100		
					5.04	30		
					4.43	80		
					3.06	30		
					2.54	50		
					2.23	10		
					1.68	30		
					1.49	80		
					1.29	30		
					1.24	20		
					1.12	10		
					1.03	10		
					0.97	10		
					0.87	10		
					Diffuse Pattern			

(Al_{1.51}Fe_{.13}Mg_{.40}) (Al_{.07}Si_{3.43})

O₁₀(OH)₂ (Na_{.02}Ca_{.18})

Montmorillonite

Rad: Cu Kα

Filter: Nickel

Macon, Georgia - Kaolinite

Diffraction Pattern

Intensity	Order of Intensity	Interplanar Spacing (Å)
469	1	7.25
48	4	3.94
161	2	3.59
36	5	2.55
35		2.33#
16		2.02#
72	3	1.67
28	8	1.49
32	6	1.43
28	9	1.34
32	7	1.31
18	10	1.28
48		1.22#

These lines are the Hannawalt Index for pure Aluminum Metal from the sample holder and are therefore excluded from the order of intensity count.

ASTM Card for Kaolinite

d	3.59	7.2	4.45	d in Å λ = .708	I/I ₁	d in Å λ = .708	I/I ₁
I/I ₁	1.0	0.80	0.80	7.20	0.80	1.80	0.80
I	12.5	10	10	4.45	0.80	1.67	0.40
Al ₂ Si ₂ O ₆ (OH) ₄				4.30	0.64	1.54	0.16
				4.20	0.48	1.49	0.56
				4.04	0.32	1.31	0.08
				3.59	1.00	1.289	0.16
Kaolinite				2.56	0.48	1.24	0.16
				2.50	0.48		
				2.34	0.80		
Z				1.99	0.32		
Ao	bo	Co		1.90	0.08		
A		C		1.85	0.08		
D							
n	w	c					

Bedford, Indiana - Halloysite

Diffraction Pattern

Intensity	Order of Intensity	Interplanar Spacing (Å)
85	1	7.25
10	8	4.87
28	4	4.38
20	6	3.59
5	11	2.54
14		2.34#
3	12	2.22
4		2.02#
49	2	1.48
21	5	1.43
10	9	1.40
8	10	1.34
3	13	1.28
20	7	1.23
70		1.22#
30	3	1.17

These lines are the Hannawalt Index for pure Aluminum Metal from the sample holder and are therefore excluded from the order of intensity count.

ASTM Card for Halloysite

d	4.44	7.21	3.59	7.21	d = Å	I/I ₁	d in Å	I/I ₁
					λ = 1.539			
I/I ₁	100	60	50	60	7.21	60		
					4.44	100		
Al ₂ Si ₂ O ₅ (OH ₄)					3.59	50		
Halloysite					2.56	25		
Rad: CuKα					2.50	25		
Filter: Nickel					2.33	12		
					2.24	12		
					1.49	40		
					1.24	6		
					Poor Patterns with unsatisfactory lines and bands			

APPENDIX E

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Thesis

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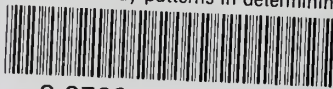
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